Supporting Information

Cu-Catalyzed N-S Bond Coupling between Hydroxylamines and Sulfinic Acids: Rapid Access to *N*-Arylsulfonamides

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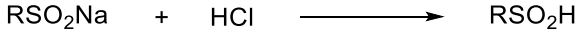
General:

All reagents were purchased from commercial suppliers and used without further purification. Silicone oil baths were used as the heat sources for all reactions that required heating. Flash chromatography was carried out with silica gel (200-300 mesh). Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. ^{1}H NMR (400 MHz) and ^{13}C NMR (101 MHz) spectra were recorded in $CDCl_3$. Chemical shifts (δ) are reported in ppm using TMS as internal standard and spin-spin coupling constants (*J*) are given in Hz. Proton and carbon multiplicity are recorded as singlet (s), doublet (d), triplet (t), quartet (q), quintet (quin), sextet (sex) septet (sep) multiplet (m) and broad (br). Melting points were measured by means of a micro melting point apparatus, SGWX-4B, supplied by Shanghai Yidian Physical Optics Instrument Co., Ltd. High resolution mass spectra were performed on Bruker Daltonics APEXII 47e Specifications. HRMS data were measured by means of the ESI technique on Fourier transform ion cyclotron resonance mass analyzer. Data collections for crystal structure were performed at 293 K using MoKα radiation on a Bruker Smart Apex II diffractometer.

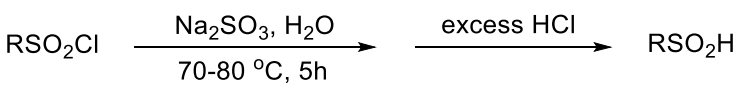
Substrates preparation

General procedure for the synthesis of sulfinic acids (1a-b, 1d, 1l).



The sodium sulfinate (10 mmol) was acidified by excess concentrated HCl at 0 °C and then the mixture was extracted by Et_2O. After dried by anhydrous Na_2SO_4, the solvent was removed under vacuum at 0 °C to afford the product. [1]

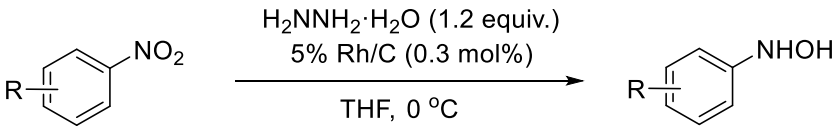
General procedure for the synthesis of sulfinic acids (1c, 1e-k).



To a flame dried 100 mL round bottom flask, sulfonyl chloride (10 mmol), anhydrous sodium sulfite (30 mmol) and H_2O (20 mL) were added in sequence. The reaction mixture was kept at 70-80 °C for 5 h. After the reaction was complete, the mixture was

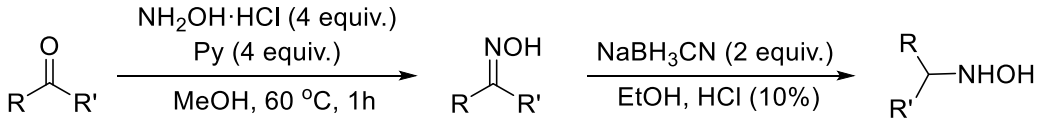
washed with chloroform. Next, the water phase was acidified by excess concentrated HCl at 0 °C and then extracted by Et_2O. After dried by anhydrous Na_2SO_4, the organic solvent was removed under vacuum at 0 °C to afford the product. [2]

General procedure for the synthesis of arylhydroxylamines.



Under Ar atmosphere, a suspension of nitrobenzene (5 mmol, 1 equiv.) and 5% Rh/C (0.30 mol% Rh) in THF (20 mL) was cooled to 0 ℃. Hydrazine monohydrate (6 mmol, 1.2 equiv.) was added dropwise. The reaction mixture was stirred at 0 ℃ for 1 h and then slowly warmed up to room temperature and stirred at room temperature for 4 h. The reaction mixture was filtered through a short pad of celite and concentrated *in vacuo.* Recrystallization from CH_2Cl_2/hexanes at –20 °C afforded the corresponding arylhydroxylamines. [3]

General procedure for the synthesis of alkylhydroxylamines.



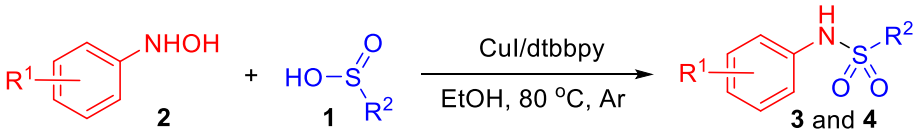
Ketone/aldehyde (10 mmol), hydroxylamine hydrochloride (4 equiv.), pyridine (4 equiv.) in CH_3OH (20 mL) were stirred at 60 °C for 1 h. After completion of the reaction, the solvent was removed under reduced pressure. The mixture was diluted with H_2O (30 mL) and extracted with EtOAc (3 × 20 mL). The organic layer was washed with 1 M HCl, dried over anhydrous Na_2SO_4, and filtered. The solvent was removed under reduced pressure. All the obtained products were used for the next step without further purification. [4]

A mixture of oxime (10 mmol) and sodium cyanoborohydride (20 mmol, 2 equiv.) in ethanol (30 mL) was cooled to 0 ˚C. To this mixture was added 10% HCl (aq) (35 mmol) over a period of 20 minutes, during which time the temperature was maintained at 0 ˚C. The reaction mixture was stirred for a further 1 hour at 0 ˚C and then at room temperature for 2 hours. After this time the mixture was diluted with water (150 mL) and adjusted to

pH 10 by the addition of 20 % NaOH (aq) to precipitate the product. The precipitate formed was filtered, washed with water, and dried *in vacuo*, to give the desired product. [5]

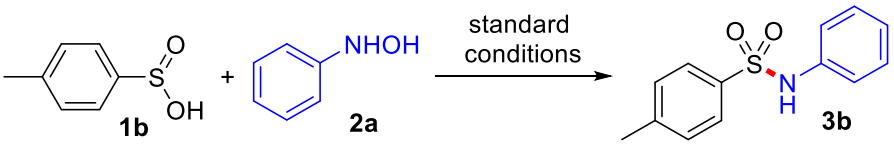
General experimental procedure

Typical experimental procedure for the synthesis of *N*-arylsulfamides 3 and 4.



A 25 mL oven-dried reaction tube were charged with sulfinic acids **1** (0.2 mmol), hydroxylamines **2** (0.5 mmol, 2.5 equiv.), CuI (0.04 mmol, 20 mol%), dtbbpy (0.04 mmol, 20 mol%) and 2 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing. Then the mixture was stirred for 12 h at 80 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel to give the corresponding products **3** and **4**.

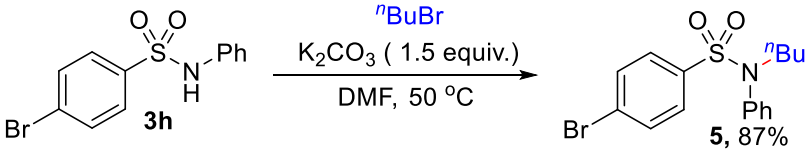
Gram-scale synthesis of 3b



A 100 mL oven-dried reaction tube were charged with sulfinic acid **1b** (5 mmol), hydroxylamine **2a** (7.5 mmol, 2.5 equiv.), CuI (20 mol%), dtbbpy (20 mol%) and 50 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing. Then the mixture was stirred for 12 h at 80 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel to give the desired product **3b** (1.05 g, 85%).

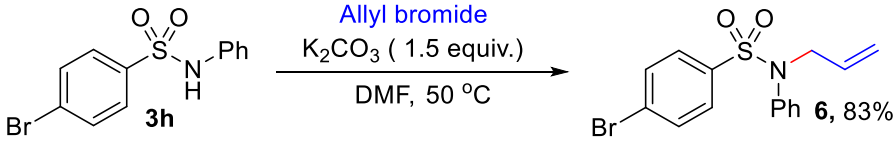
Follow-up transformations of 3h

General procedure for the synthesis of 4-Bromo-*N*-butyl-*N*-phenylbenzenesulfonamid- e 5.



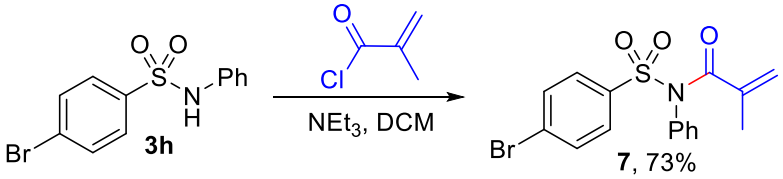
A 25 mL oven-dried reaction tube were charged with sulfonamide **3h** (0.2 mmol), K_2CO_3 (0.3 mmol,1.5 equiv.), ^{n}BuBr (0.4 mmol, 2 equiv.), and 3 mL DMF. The mixture was stirred for 12 h at 50 °C and quenched with H_2O (10 mL), extracted with EA (15 mL x 3). The combined organic layer was washed with brine (10 mL × 3), dried over Na_2SO_4, and concentrated. The residue was purified by flash chromatography on a silica gel using petroleum ether and ethyl acetate as the eluent to give the desired product **5** in 87% yield. [6]

General procedure for the synthesis of *N*-allyl-4-bromo-*N*-phenylbenzenesulfonamide 6.



A 25 mL oven-dried reaction tube were charged with sulfonamide **3h** (0.2 mmol), K_2CO_3 (0.3 mmol,1.5 equiv.), allyl bromide (0.4 mmol, 2 equiv.), and 3 mL DMF. The mixture was stirred for 12 h at 50 °C and quenched with H_2O (10 mL), extracted with EA (15 mL x 3). The combined organic layer was washed with brine (10 mL × 3), dried over Na_2SO_4, and concentrated. The residue was purified by flash chromatography on a silica gel using petroleum ether and ethyl acetate as the eluent to give the desired product **6** in 83% yield.[6]

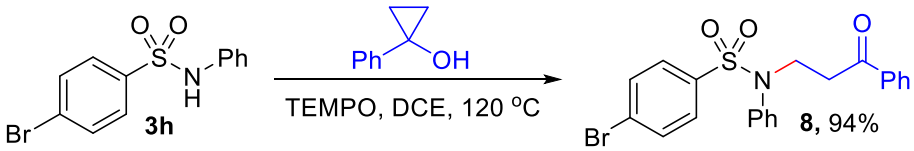
General procedure for the synthesis of *N*-((4-bromophenyl)sulfonyl)-*N*-phenylmeth-acrylamide 7.



A 25 mL oven-dried reaction tube were charged with sulfonamide **3h** (0.2 mmol), Et_3N (0.6 mmol) in 5 mL of DCM were stirred in ice bath. Then methacryloyl chloride (0.24

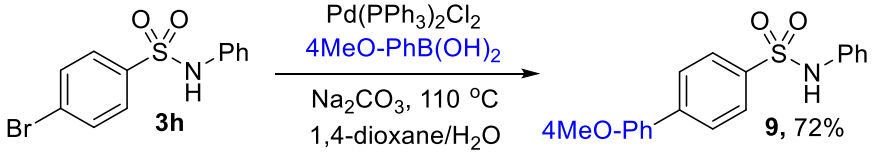
mmol, 1.2 equiv.) was added slowly and the mixture was stirred at room temperature. The progress of the reaction was monitored by thin-layer chromatography (TLC). The reaction was quenched by H_2O (10 mL), and the aqueous layer was extracted with DCM (3 × 10 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography on a silica gel using petroleum ether and ethyl acetate as the eluent to give the desired product **7** in 73% yield.[7]

General procedure for the synthesis of 4-bromo-*N*-(3-oxo-3-phenylpropyl)-*N*-phenyl- benzenesulfonamide 8.



A 25 mL oven-dried reaction pressure tube were charged with sulfonamide **3h** (0.2 mmol), cyclopropanol (0.5 mmol, 2.5 equiv.), TEMPO (0.4 mmol, 2 equiv.) and DCE (2 mL). The tube was then sealed and the mixture was stirred for 48 h at 120 ℃. After completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether) to give the corresponding product **8** in 94% yield. [8]

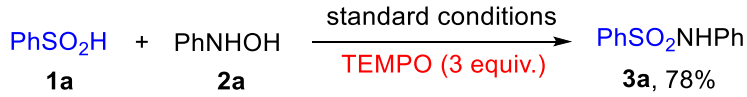
General procedure for the synthesis of 4'-methoxyl-*N*-phenyl-[1,1'-biphenyl]-4-sulfonamide 9.



A 25 mL oven-dried reaction pressure tube were charged with sulfonamide **3h** (0.2 mmol), 1,4-dioxane (3 mL) and H_2O (1 mL). After compound **3h** was stirred and dissolved, 4-MeO-PhB$(OH)_2$ (0.2 mmol, 1 equiv.), Na_2CO_3 (0.4 mmol, 2 equiv.) and $Pd(PPh_3)_2Cl_2$ (0.01 mmol, 5 mol%) were added in sequence. The reaction was flushed with argon and heated to 110 °C for 16 h. After completion of the reaction, the reaction quenched with H_2O (10 mL), extracted with EA (15 mL x 3). The combined organic layer was washed with brine (10 mL × 3), dried over Na_2SO_4, and concentrated. The

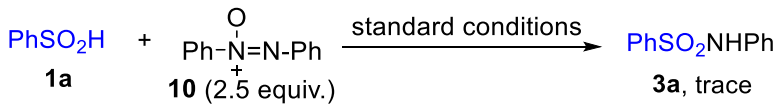
residue was purified by flash chromatography on a silica gel using petroleum ether and ethyl acetate as the eluent to give the desired product **9** in 72% yield. [9]

Control experiments

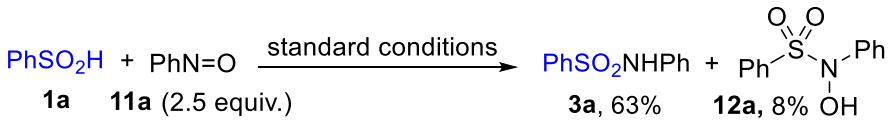


A 25 mL oven-dried reaction tube were charged with sulfinic acid **1a** (0.2 mmol), hydroxylamine **2a** (0.5 mmol, 2.5 equiv.), CuI (0.04 mmol, 20 mol%), dtbbpy (0.04 mmol, 20 mol%), TEMPO (0.6 mmol, 3 equiv.) and 2 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing. Then the mixture was stirred for 12 h at 80 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel to give the corresponding product **3a** in 78% yield.

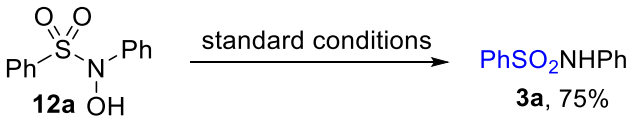
A 25 mL oven-dried reaction tube were charged with sulfinic acid **1a** (0.2 mmol), hydroxylamine **2a** (0.5 mmol, 2.5 equiv.), CuI (0.04 mmol, 20 mol%), dtbbpy (0.04 mmol, 20 mol%), BHT (0.6 mmol, 3 equiv.) and 2 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing. Then the mixture was stirred for 12 h at 80 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel to give the desired product **3a** in 83% yield.



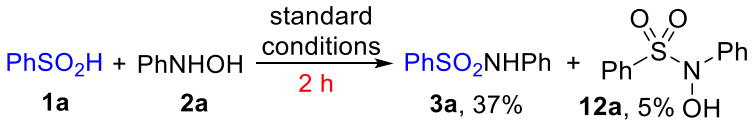
A 25 mL oven-dried reaction tube were charged with sulfinic acid **1a** (0.2 mmol), azoxybenzene **10** (0.5 mmol, 2.5 equiv.), CuI (0.04 mmol, 20 mol%), dtbbpy (0.04 mmol, 20 mol%) and 2 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing. Then the mixture was stirred for 12 h at 80 °C. Trace amounts of **3a** was detected after TLC (Thin Layer Chromatography) detection.



A 25 mL oven-dried reaction tube were charged with sulfinic acid **1a** (0.2 mmol), nitrosobenzene **11a** (0.5 mmol, 2.5 equiv.), CuI (0.04 mmol, 20 mol%), dtbbpy (0.04 mmol, 20 mol%) and 2 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing. Then the mixture was stirred for 12 h at 80 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel to give the desired products **3a** in 63% yield, accompanied by 8% of *N*-hydroxy-*N*-phenylbenzenesulfonamide **12a**.



A 25 mL oven-dried reaction tube were charged with **12a** (0.2 mmol), CuI (0.04 mmol, 20 mol%), dtbbpy (0.04 mmol, 20 mol%) and 2 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing. Then the mixture was stirred for 12 h at 80 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel to give the desired product **3a** in 75% yield.

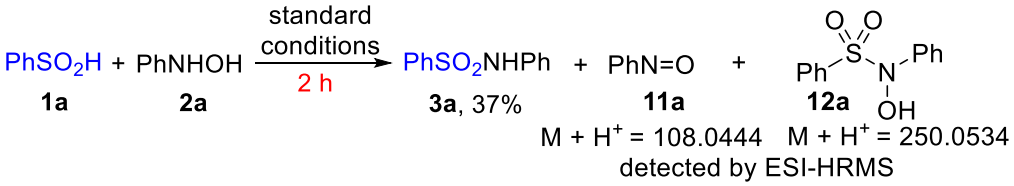


A 25 mL oven-dried reaction tube were charged with sulfinic acid **1a** (0.2 mmol), hydroxylamine **2a** (0.5 mmol, 2.5 equiv.), CuI (0.04 mmol, 20 mol%), dtbbpy (0.04 mmol, 20 mol%) and 2 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing and then the mixture was stirred for 2 h at 80 °C. The solvent was removed under vacuo and the residue was purified with chromatography column on silica gel to give the desired product **3a** in 37% yield and the intermediate **12a** in 5% yield.

HRMS analysis for reaction intermediates.

A 25 mL oven-dried reaction tube were charged with sulfinic acid **1a** (0.2 mmol), hydroxylamine **2a** (0.5 mmol, 2.5 equiv.), CuI (0.04 mmol, 20 mol%), dtbbpy (0.04

mmol, 20 mol%) and 2 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing and then the mixture was stirred in an oil bath for 2 h at 80 °C. Subsequently, the reaction was cooled to room temperature and diluted with DCM prior to the injection into the high-resolution mass spectrometer. The compound **11a**: HRMS (ESI) m/z $[M + H]^+$ Calcd for 108.0449, found 108.0444; the compound **12a**: HRMS (ESI) m/z $[M + H]^+$ Calcd for 250.0538, found 250.0534.

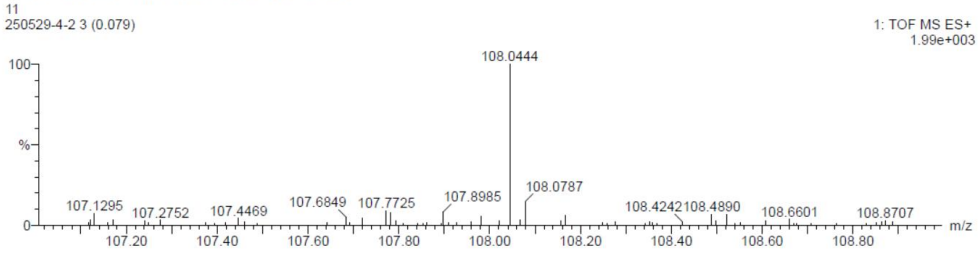


Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
71 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 6-6 H: 6-6 N: 0-100 O: 0-100 Na: 0-7

11
250529-4-2 3 (0.079)



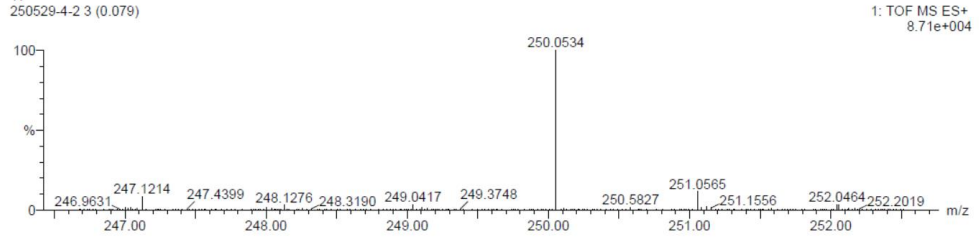
Minimum:			-1.5					
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
108.0444	108.0449	-0.5	-4.6	4.5	231.6	n/a	n/a	C6 H6 N O

Elemental Composition Report **Page 1**

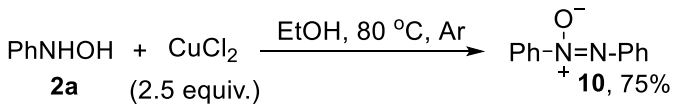
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 5

Monoisotopic Mass, Even Electron Ions
1333 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 12-12 H: 12-12 N: 0-100 O: 0-100 Na: 0-7 S: 0-2

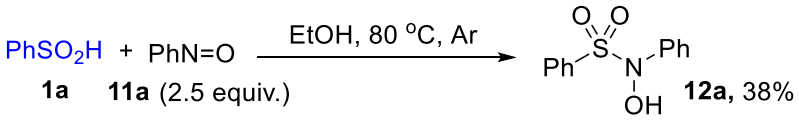
11
250529-4-2 3 (0.079)



Minimum:			-1.5					
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
250.0534	250.0538	-0.4	-1.6	7.5	1285.6	n/a	n/a	C12 H12 N O3 S



A 25 mL oven-dried reaction tube were charged with hydroxylamine **2a** (0.2 mmol), $CuCl_2$ (0.5 mmol, 2.5 equiv.) and 6 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing. The mixture was stirred for 12 h at 80 °C and then the solvent was removed under vacuo. Next, the residue was purified with chromatography column on silica gel to give the azoxybenzene **10** in 75% yield.



A 25 mL oven-dried reaction tube were charged with sulfinic acid **1a** (0.2 mmol), nitrosobenzene **11a** (0.5 mmol, 2.5 equiv.) and 2 mL EtOH. The reaction tube was subjected to three argon purges prior to sealing. Then the mixture was stirred for 12 h at 80 °C. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel to give the *N*-hydroxy-*N*-phenylbenzenesulfonamide **12a** in 38% yield.

Crystal Data for Compound 3f

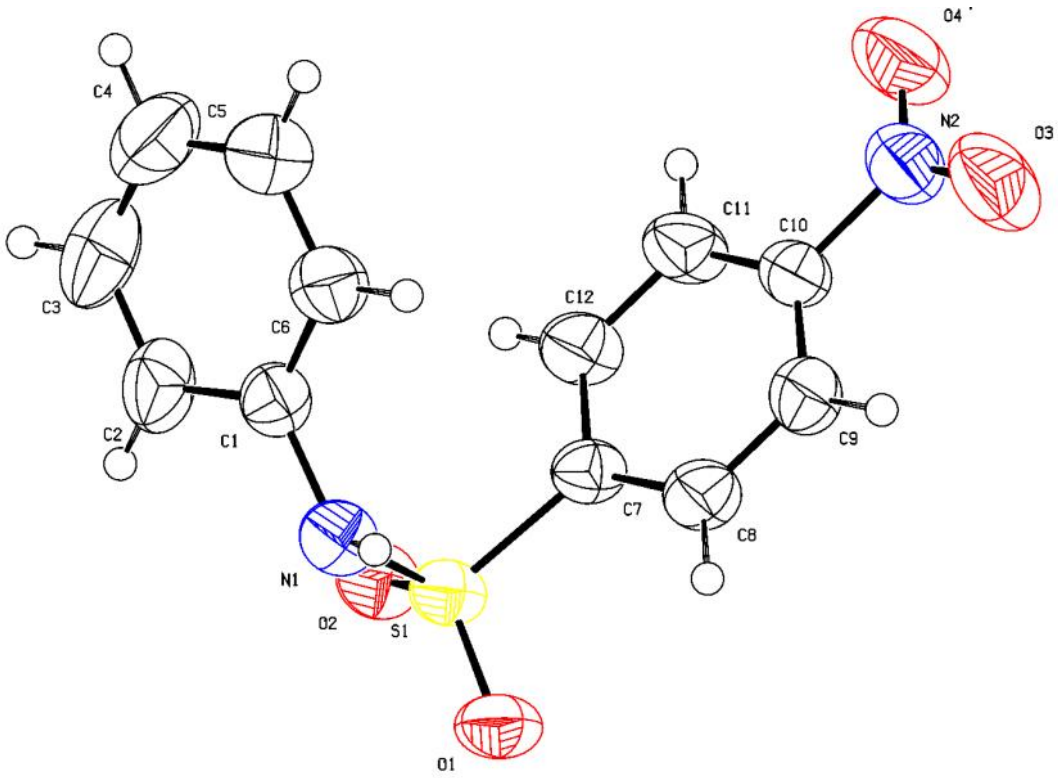


Figure S1. ORTEP plot of compound **3f**. Thermal ellipsoids are given at the 50% probability level

In a 2 mL vial, 20 mg of **3f** was dissolved in 0.5 mL of dichloromethane. Then, 1 mL of *n*-hexane was added, and the mixture was sonicated for 3 minutes. After sonication, the vial was placed in a light-protected area and slowly evaporated at room temperature to obtain crystal.

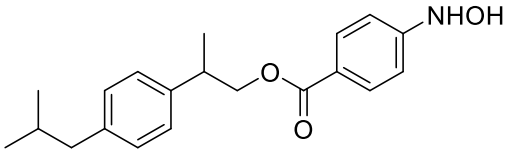
Table S1. The crystal data and refinement results of compound **3f**

Compound number	**3f**
Formula	$C_{12}H_{10}N_2O_4S$
Fw	278.28
Temp	273.15
Crystal system	monoclinic
Space group	Cc
a Å	5.1939(3)
b Å	12.7888(9)
c Å	18.6760(13)
α°	90.00
β°	93.318(2)
γ°	90.00
V $Å^3$	1238.45(14)
Z	4
Density(calcd) g cm^{-3}	1.498
Absorb.coeff. mm^{-1}	0.273
F(000)	580.0
Index ranges	$-5 \leq h \leq 6$ $-15 \leq k \leq 15$ $-22 \leq l \leq 22$

refln./restr./param.	2103/2/172
GOF	1.108
[$I > 2\sigma(I)$]	R_1 = 0.0331 Rw_2 = 0.0777
CCDC number	2281475

Analytical data for substrates:

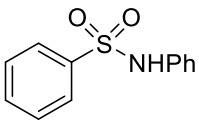
2-(4-Isobutylphenyl)propyl 4-(hydroxyamino)benzoate (2n)



The product was obtained by Recrystallization from CH_2Cl_2/hexanes at –20 °C, White solid; (1.61 g, 98%); mp: 109–110 °C; R_f = 0.19 (hexanes/ethyl acetate 3: 1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, J = 8.4 Hz, 2H), 7.10 (d, J = 8.0 Hz, 2H), 7.01 (d, J = 8.0 Hz, 2H), 6.89 (brs, 1H), 6.85 (d, J = 8.4 Hz, 2H), 5.89 (brs, 1H), 4.31–4.21 (m, 2H), 3.11 (sex, J = 7.2 Hz, 1H), 2.36 (d, J = 7.2 Hz, 2H), 1.76 (sep, J = 6.8 Hz, 1H), 1.28 (d, J = 6.8 Hz, 3H), 0.81 (d, J = 6.8 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 166.6, 154.0, 140.4, 140.0, 130.9, 129.2, 127.0, 123.3, 112.9, 69.8, 45.0, 38.6, 30.2, 22.4, 22.3, 18.0; ESI-HRMS (ESI, m/z): Calcd for $C_{20}H_{26}NO_3$, $[M + H]^+$: 328.1907, found 328.1903.

Analytical data for products:

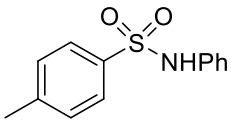
N-Phenylbenzenesulfonamide (3a)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), White solid; (38 mg, 82%); mp: 104–105 °C; R_f = 0.23 (hexanes/ethyl acetate 5: 1);[10a] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82–7.80 (m, 2H), 7.51 (t, J = 7.6 Hz, 1H), 7.42 (t, J = 7.6 Hz, 2H), 7.34 (brs, 1H), 7.24–7.20 (m, 2H), 7.09 (t, J = 7.2 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 138.9, 136.4, 133.0, 129.3, 129.0, 127.2, 125.3, 121.6; ESI-HRMS

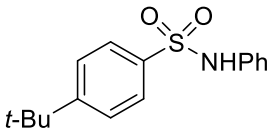
(ESI, m/z): Calcd for $C_{12}H_{12}NO_2S$, $[M + H]^+$: 234.0589, found 234.0592.

4-Methyl-N-phenylbenzenesulfonamide (3b)



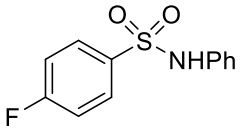
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), White solid; (46 mg, 93%); mp: 116–118 ^oC; R_f = 0.25 (hexanes/ethyl acetate 5: 1);[10b] ^1H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, J = 8.0 Hz, 2H), 7.26–7.20 (m, 4H), 7.15 (brs, 1H), 7.10–7.07 (m, 3H), 2.36 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 143.8, 136.5, 136.0, 129.6, 129.2, 127.2, 125.2, 121.4, 21.5; ESI-HRMS (ESI, m/z): Calcd for $C_{13}H_{14}NO_2S$, $[M + H]^+$: 248.0745, found 248.0739.

4-(tert-Butyl)-N-phenylbenzenesulfonamide (3c)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), Yellow solid; (52 mg, 90%); mp: 166–170 ^oC; R_f = 0.32 (hexanes/ethyl acetate 5: 1);[10c] ^1H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, J = 8.4 Hz, 2H), 7.43 (d, J = 8.4 Hz, 2H), 7.23 (t, J = 7.6 Hz, 2H), 7.11–7.09 (m, 4H), 1.29 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 156.8, 136.6, 136.1, 129.3, 127.0, 126.0, 125.1, 121.2, 35.1, 31.0; ESI-HRMS (ESI, m/z): Calcd for $C_{16}H_{20}NO_2S$, $[M + H]^+$: 290.1215, found 290.1221.

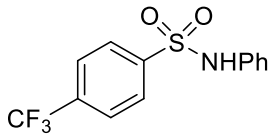
4-Fluoro-N-phenylbenzenesulfonamide (3d)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), Yellow solid; (34 mg, 68%); mp: 108–112 ^oC; R_f = 0.31 (hexanes/ethyl acetate 5: 1); [10d] ^1H NMR (400 MHz, $CDCl_3$): δ 7.81–7.76 (m, 2H), 7.27–7.23 (m, 2H), 7.16–7.06 (m, 5H), 5.13 (brs, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 165.2 (d, $^1J_{C\text{-}F}$ = 254 Hz), 136.1, 134.9 (d, $^4J_{C\text{-}F}$ = 3 Hz), 130.0 (d, $^3J_{C\text{-}F}$ = 10 Hz), 129.4, 125.8, 122.0, 116.3 (d, $^2J_{C\text{-}F}$ = 23 Hz); ^{19}F NMR (376 MHz, $CDCl_3$): δ -104.4; ESI-HRMS (ESI, m/z): Calcd for

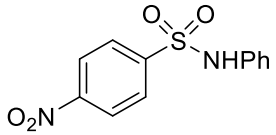
$C_{12}H_{11}FNO_2S$, $[M + H]^+$: 252.0495, found 252.0492.

N-Phenyl-4-(trifluoromethyl)benzenesulfonamide (3e)



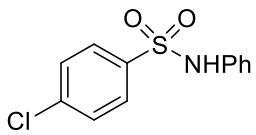
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), White solid; (42 mg, 69%); mp: 89–92 °C; R_f = 0.27 (hexanes/ethyl acetate 5: 1);[10e] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, J = 8.4 Hz, 2H), 7.70 (d, J = 8.0 Hz, 2H), 7.29–7.26 (m, 2H), 7.17 (t, J = 7.2 Hz, 1H), 7.08 (d, J = 7.6 Hz, 2H), 6.84 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 142.5, 135.6, 134.7 (q, $^2J_{C\text{-}F}$ = 33 Hz), 129.6, 127.7, 126.2 (q, $^3J_{C\text{-}F}$ = 4 Hz), 126.1, 123.1 (q, $^1J_{C\text{-}F}$ = 272 Hz), 122.2; ^{19}F NMR (376 MHz, $CDCl_3$): δ -63.2; ESI-HRMS (ESI, m/z): Calcd for $C_{13}H_{10}F_3NO_2SNa$, $[M + Na]^+$: 324.0282, found 324.0273.

4-Nitro-N-phenylbenzenesulfonamide (3f)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), Yellow solid; (32 mg, 58%); mp: 135–136 °C; R_f = 0.21 (hexanes/ethyl acetate 5: 1);[10a] ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.28 (d, J = 8.8 Hz, 2H), 7.93 (d, J = 8.8 Hz, 2H), 7.30–7.26 (m, 2H), 7.19 (t, J = 7.2 Hz, 1H), 7.08 (d, J = 7.6 Hz, 2H), 6.83 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 150.2, 144.6, 135.3, 129.7, 128.5, 126.5, 124.3, 122.4; ESI-HRMS (ESI, m/z): Calcd for $C_{12}H_{11}N_2O_4S$, $[M + H]^+$: 279.0440, found 279.0439.

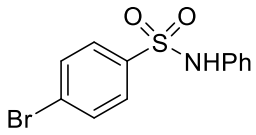
4-Chloro-N-phenylbenzenesulfonamide (3g)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), Yellow solid; (45 mg, 85%); mp: 94–99 °C; R_f = 0.36 (hexanes/ethyl acetate 5: 1);[10f] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, J = 8.4 Hz, 2H), 7.40 (d, J = 8.4 Hz, 2H), 7.26 (t, J = 7.6 Hz, 2H), 7.15 (t, J = 7.2 Hz, 1H), 7.07 (d, J = 7.6 Hz, 2H), 6.79 (s, 1H);

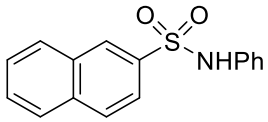
^{13}C NMR (101 MHz, $CDCl_3$): δ 139.6, 137.4, 135.9, 129.5, 129.3, 128.6, 125.9, 122.0; ESI-HRMS (ESI, m/z): Calcd for $C_{12}H_{11}ClNO_2S$, $[M + H]^+$: 268.0199, found 268.0202.

4-Bromo-N-phenylbenzenesulfonamide (3h)



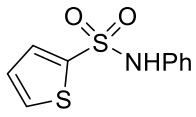
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 20 : 1), Yellow solid; (45 mg, 72%); mp: 110–112 ^{o}C; R_f = 0.38 (hexanes/ethyl acetate 5: 1);[10g] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, J = 8.8 Hz, 2H), 7.56 (d, J = 8.4 Hz, 2H), 7.26 (t, J = 7.6 Hz, 2H), 7.14 (t, J = 7.6 Hz, 1H), 7.07 (d, J = 7.6 Hz, 2H), 6.91 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 137.9, 135.9, 132.3, 129.5, 128.7, 128.1, 125.9, 122.0; ESI-HRMS (ESI, m/z): Calcd for $C_{12}H_{11}BrNO_2S$, $[M + H]^+$: 311.9694, found 311.9693.

N-Phenylnaphthalene-2-sulfonamide (3i)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 20 : 1), Yellow solid; (45 mg, 80%); mp: 131–133 ^{o}C; R_f = 0.44 (hexanes/ethyl acetate 5: 1);[10h] ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.36 (d, J = 1.2, 1H), 7.88 (t, J = 8.4, 3H), 7.74 (dd, J = 2.0 Hz, J = 8.8 Hz, 1H); 7.64–7.56 (m, 2H), 7.23–7.19 (m, 2H), 7.11–7.07 (m, 2H), 6.79 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.4, 135.8, 134.8, 132.0, 129.4, 129.3, 129.2, 128.9, 127.8, 127.5, 125.3, 122.2, 121.5; ESI-HRMS (ESI, m/z): Calcd for $C_{16}H_{14}NO_2S$, $[M + H]^+$: 284.0745, found 284.0747.

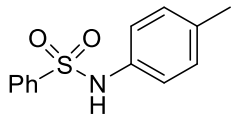
N-Phenylthiophene-2-sulfonamide (3j)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 5 : 1), Yellow solid; (31 mg, 65%); mp: 89–92 ^{o}C; R_f = 0.41 (hexanes/ethyl acetate 3: 1);[10i] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (dd, J = 1.2 Hz, J = 5.2 Hz, 1H), 7.50 (dd, J = 1.2 Hz, J = 4.0 Hz, 1H), 7.30–7.26 (m, 2H), 7.18–7.13 (m, 3H), 7.00 (dd, J = 4.0 Hz, J =

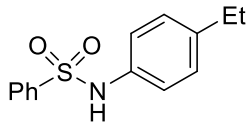
4.8 Hz, 1H), 6.89 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 139.3, 136.1, 132.9, 132.4, 129.4, 127.3, 125.8, 121.9; ESI-HRMS (ESI, m/z): Calcd for $C_{10}H_{10}NO_2S_2$, $[M + H]^+$: 240.0153, found 240.0148.

N-(p-Tolyl)benzenesulfonamide (4b)



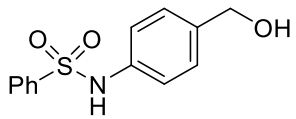
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), Yellow solid; (35 mg, 71%); mp: 117–119 ^{o}C; R_f = 0.30 (hexanes/ethyl acetate 5: 1);[11a] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, J = 7.6 Hz, 2H), 7.52 (t, J = 7.6 Hz, 1H), 7.42 (t, J = 7.6 Hz, 2H), 7.03 (d, J = 8.4 Hz, 2H), 6.95 (d, J = 8.0 Hz, 2H), 6.83 (s, 1H), 2.26 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 139.0, 135.6, 133.5, 132.9, 129.8, 128.9, 127.2, 122.4, 20.8; ESI-HRMS (ESI, m/z): Calcd for $C_{13}H_{14}NO_2S$, $[M + H]^+$: 248.0745, found 248.0745.

N-(4-Ethylphenyl)benzenesulfonamide (4c)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), Brown solid; (35 mg, 67%); mp: 73–77 ^{o}C; R_f = 0.32 (hexanes/ethyl acetate 5: 1);[11b] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, J = 8.0 Hz, 2H), 7.52 (t, J = 7.6 Hz, 1H), 7.42 (t, J = 8.0 Hz, 2H), 7.06–7.03 (m, 3H), 6.99 (d, J = 8.4 Hz, 2H), 2.56 (q, J = 7.6 Hz, 2H), 1.16 (t, J = 7.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.7, 139.0, 133.8, 132.8, 128.9, 128.6, 127.2, 122.3, 28.2, 15.4; ESI-HRMS (ESI, m/z): Calcd for $C_{14}H_{16}NO_2S$, $[M + H]^+$: 262.0902, found 262.0905.

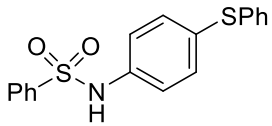
N-(4-(Hydroxymethyl)phenyl)benzenesulfonamide (4d)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 5 : 1), Yellow oil; (41 mg, 77%); R_f = 0.25 (hexanes/ethyl acetate 3: 1);[11c] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, J = 7.6 Hz, 2H), 7.51 (t, J = 7.6 Hz, 1H), 7.41 (t, J = 7.6 Hz,

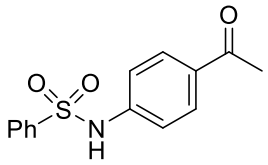
2H), 7.32 (s, 1H), 7.19 (d, J = 8.0 Hz, 2H), 7.03 (d, J = 8.0 Hz, 2H), 4.58 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 138.9, 137.9, 135.7, 133.0, 129.0, 128.0, 127.2, 121.7, 64.6; ESI-HRMS (ESI, m/z): Calcd for $C_{13}H_{13}NO_3SNa$, $[M + Na]^+$: 286.0514, found 286.0518.

N-(4-(Phenylthio)phenyl)benzenesulfonamide (4e)



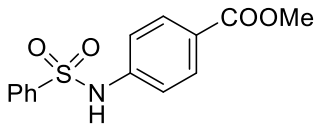
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 5 : 1), Grey solid; (58 mg, 85%); mp: 89–93 ^{o}C; R_f = 0.46 (hexanes/ethyl acetate 3: 1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, J = 7.2 Hz, 2H), 7.56 (t, J = 7.6 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 7.30–7.20 (m, 7H), 7.00 (d, J = 8.4 Hz, 2H), 6.76 (s, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 138.9, 135.5, 135.4, 133.2, 132.6, 132.2, 130.8, 129.2, 129.1, 127.2, 127.1, 122.3; ESI-HRMS (ESI, m/z): Calcd for $C_{18}H_{16}NO_2S_2$, $[M + H]^+$: 342.0622, found 342.0618.

N-(4-Acetylphenyl)benzenesulfonamide (4f)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 5 : 1), White solid; (52 mg, 95%); mp: 125–128 ^{o}C; R_f = 0.51 (hexanes/ethyl acetate 2: 1);[11d] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (t, J = 7.2 Hz, 4H), 7.57–7.55 (m, 1H), 7.49–7.42 (m, 3H), 7.18 (d, J = 8.0 Hz, 2H), 2.54 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 196.8, 141.0, 138.8, 133.5, 133.4, 130.0, 129.3, 127.2, 119.1, 26.4; ESI-HRMS (ESI, m/z): Calcd for $C_{14}H_{14}NO_3S$, $[M + H]^+$: 276.0694, found 276.0696.

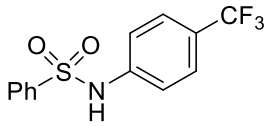
Methyl 4-(phenylsulfonamido)benzoate (4g)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 5 : 1), White solid; (46 mg, 79%); mp: 145–150 ^{o}C; R_f = 0.40 (hexanes/ethyl acetate 3:

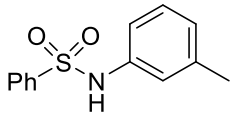
1);[11b] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, J = 8.4 Hz, 2H), 7.84 (d, J = 7.6 Hz, 2H), 7.56 (t, J = 7.6 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 7.14 (d, J = 8.8 Hz, 2H), 7.04 (s, 1H), 3.87 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 166.3, 140.7, 138.8, 133.4, 131.1, 129.2, 127.2, 126.4, 119.2, 52.1; ESI-HRMS (ESI, m/z): Calcd for $C_{14}H_{14}NO_4S$, $[M + H]^+$: 292.0644, found 292.0647.

N-(4-(Trifluoromethyl)phenyl)benzenesulfonamide (4h)



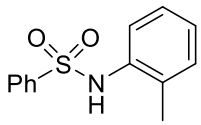
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 5 : 1), Yellow solid; (44 mg, 73%); mp: 92–94 ^{o}C; R_f = 0.40 (hexanes/ethyl acetate 3: 1);[11b] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88–7.85 (m, 2H), 7.58 (t, J = 7.6 Hz, 1H), 7.54 (s, 1H), 7.50–7.46 (m, 4H), 7.20 (d, J = 8.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 139.7, 138.6, 133.5, 129.3, 127.2, 126.8 (q, $^2J_{C\text{-}F}$ = 33 Hz), 126.6 (q, $^3J_{C\text{-}F}$ = 4 Hz), 123.8 (q, $^1J_{C\text{-}F}$ = 271 Hz), 119.8; ^{19}F NMR (376 MHz, $CDCl_3$): δ -62.3; ESI-HRMS (ESI, m/z): Calcd for $C_{13}H_{10}F_3NO_2SNa$, $[M + Na]^+$: 324.0282, found 324.0290.

N-(m-Tolyl)benzenesulfonamide (4i)



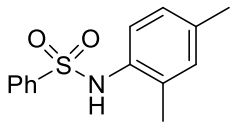
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 5 : 1), Grey brown solid; (37 mg, 74%); mp: 87–89 ^{o}C; R_f = 0.47 (hexanes/ethyl acetate 3: 1);[11b] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, J = 7.6 Hz, 2H), 7.52 (t, J = 7.6 Hz, 1H), 7.43 (t, J = 7.6 Hz, 2H), 7.15 (s, 1H), 7.09 (t, J = 8.0 Hz, 1H), 6.91–6.87 (m, 3H), 2.25 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 139.3, 139.0, 136.3, 132.9, 129.04, 128.97, 127.2, 126.2, 122.2, 118.5, 21.3; ESI-HRMS (ESI, m/z): Calcd for $C_{13}H_{14}NO_2S$, $[M + H]^+$: 248.0745, found 248.0747.

N-(o-Tolyl)benzenesulfonamide (4j)



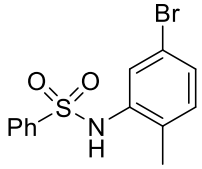
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), Red solid; (36 mg, 73%); mp: 120–124 °C; R_f = 0.36 (hexanes/ethyl acetate 5: 1);[11b] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67–7.65 (m, 2H), 7.47 (t, J = 7.6 Hz, 1H), 7.35 (t, J = 8.0 Hz, 2H), 7.23 (d, J = 8.0 Hz, 1H), 7.09–7.04 (m, 1H), 7.01 (d, J = 4.0 Hz, 2H), 6.49 (s, 1H), 1.91 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 139.6, 134.3, 132.9, 131.6, 130.8, 129.0, 127.1, 126.9, 126.4, 124.6, 17.5; ESI-HRMS (ESI, m/z): Calcd for $C_{13}H_{14}NO_2S$, $[M + H]^+$: 248.0745, found 248.0739.

N-(2,4-Dimethylphenyl)benzenesulfonamide (4k)



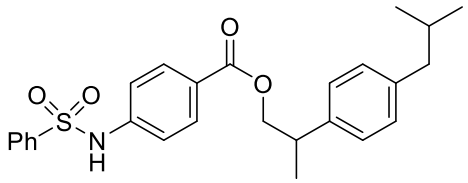
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), White solid; (43 mg, 82%); mp: 121–123 °C; R_f = 0.37 (hexanes/ethyl acetate 5: 1);[11e] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, J = 7.6 Hz, 2H), 7.55 (t, J = 7.6 Hz, 1H), 7.43 (t, J = 8.0 Hz, 2H), 7.13 (d, J = 8.0 Hz, 1H), 6.94–6.90 (m, 2H), 6.21 (s, 1H), 2.26 (s, 3H), 1.93 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 139.7, 136.5, 132.8, 132.1, 131.5, 128.9, 127.5, 127.1, 125.3, 20.8, 17.4; ESI-HRMS (ESI, m/z): Calcd for $C_{14}H_{15}NO_2SNa$, $[M + Na]^+$: 284.0721, found 284.0728.

N-(5-Bromo-2-methylphenyl)benzenesulfonamide (4l)



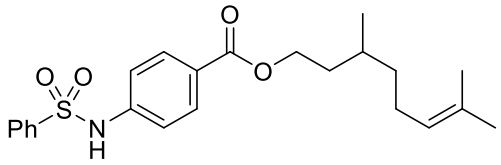
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), Red brown solid; (57 mg, 88%); mp: 125–129 °C; R_f = 0.31 (hexanes/ethyl acetate 5: 1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, J = 7.6 Hz, 2H), 7.58 (t, J = 7.6 Hz, 1H), 7.49–7.45 (m, 3H), 7.20 (dd, J = 1.6 Hz, J = 8.0 Hz, 1H), 6.95 (d, J = 8.0 Hz, 1H), 6.34 (s, 1H), 1.94 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 139.2, 135.6, 133.2, 132.0, 130.2, 129.1, 127.0, 126.9, 119.7, 17.1; ESI-HRMS (ESI, m/z): Calcd for $C_{13}H_{13}BrNO_2S$, $[M + H]^+$: 325.9850, found 325.9857.

2-(4-Isobutylphenyl)propyl 4-(phenylsulfonamido)benzoate (4n)



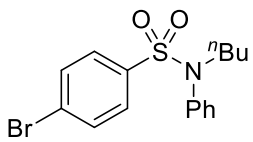
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 5: 1), Yellow solid; (61 mg, 68%); mp: 129–130 ^{o}C; R_f = 0.35 (hexanes/ethyl acetate 2: 1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, J = 8.4 Hz, 4H), 7.55 (t, J = 7.6 Hz, 1H), 7.45 (t, J = 7.6 Hz, 2H), 7.40 (brs, 1H), 7.17–7.13 (m, 3H), 7.11–7.08 (m, 3H), 4.38–4.28 (m, 2H), 3.17 (sex, J = 7.2 Hz, 1H), 2.44 (d, J = 6.8 Hz, 2H), 1.84 (sep, J = 6.8 Hz, 1H), 1.35 (d, J = 6.8 Hz, 3H), 0.88 (d, J = 6.8 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 165.8, 140.8, 140.2, 140.1, 138.8, 133.4, 131.0, 129.23, 129.20, 127.2, 127.0, 126.5, 119.1, 70.1, 45.0, 38.6, 30.2, 22.4, 22.3, 18.0; ESI-HRMS (ESI, m/z): Calcd for $C_{26}H_{29}NO_4SNa$, $[M + Na]^+$: 474.1715, found 474.1715.

3,6-Dimethylhept-5-en-1-yl 4-(phenylsulfonamido)benzoate (4o)



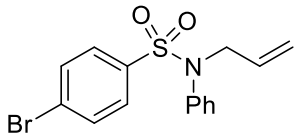
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), White solid; (43 mg, 52%); mp: 67–69 ^{o}C; R_f = 0.30 (hexanes/ethyl acetate 5: 1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92–7.90 (m, 2H), 7.85–7.83 (m, 2H), 7.56 (dt, J = 1.2 Hz, J = 7.2 Hz, 1H), 7.49–7.45 (m, 2H), 7.15–7.11 (m, 3H), 5.10–5.06 (m, 1H), 4.34–4.28 (m, 2H), 2.05–1.95 (m, 2H), 1.80–1.74 (m, 1H), 1.66 (s, 3H), 1.62–1.51 (m, 5H), 1.40–1.33 (m, 1H), 1.24–1.18 (m, 1H), 0.84 (d, J = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 165.9, 140.6, 138.8, 133.4, 131.4, 131.0, 129.2, 127.2, 126.8, 124.5, 119.2, 63.6, 36.9, 35.4, 29.5, 25.7, 25.3, 19.5, 17.6; ESI-HRMS (ESI, m/z): Calcd for $C_{23}H_{30}NO_4S$, $[M + H]^+$: 416.1896, found 416.1896.

4-Bromo-N-butyl-N-phenylbenzenesulfonamide (5)



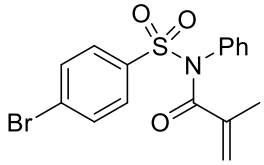
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 20 : 1), White solid; (64 mg, 87%); mp: 77–78 °C; R_f = 0.50 (hexanes/ethyl acetate 10: 1);[12a] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, J = 8.4 Hz, 2H), 7.44–7.42 (m, 2H), 7.35–7.31 (m, 3H), 7.05–7.03 (m, 2H), 3.53 (t, J = 7.2 Hz, 2H), 1.43–1.28 (m, 4H), 0.86 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 138.7, 137.3, 132.0, 129.1, 129.0, 128.7, 128.0, 127.4, 50.3, 30.2, 19.5, 13.5; ESI-HRMS (ESI, m/z): Calcd for $C_{16}H_{18}BrNO_2SNa$, $[M + Na]^+$: 390.0139, found 390.0137.

N-Allyl-4-bromo-N-phenylbenzenesulfonamide (6)



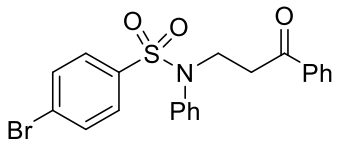
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 20 : 1), White solid; (58 mg, 83%); mp: 90–91 °C; R_f = 0.45 (hexanes/ethyl acetate 10: 1);[12b] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (dt, J = 2.0 Hz, J = 8.8 Hz, 2H), 7.46 (dt, J = 2.0 Hz, J = 8.8 Hz, 2H), 7.33–7.28 (m, 3H), 7.05–7.03 (m, 2H), 5.78–5.68 (m, 1H), 5.11–5.05 (m, 2H), 4.18 (dt, J = 1.2 Hz, J = 6.4 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 138.6, 137.4, 132.4, 132.0, 129.1, 129.0, 128.8, 128.0, 127.6, 119.1, 53.7; ESI-HRMS (ESI, m/z): Calcd for $C_{15}H_{14}BrNO_2SNa$, $[M + Na]^+$: 373.9826, found 373.9830.

N-((4-Bromophenyl)sulfonyl)-N-phenylmethacrylamide (7)



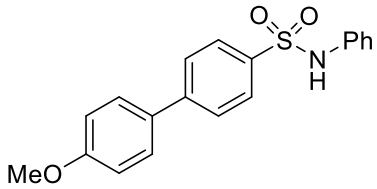
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 20 : 1), White solid; (55 mg, 73%); mp: 138–140 °C; R_f = 0.35 (hexanes/ethyl acetate 10: 1);[12c] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76–7.74 (m, 2H), 7.65–7.63 (m, 2H), 7.44–7.37 (m, 3H), 7.15–7.12 (m, 2H), 5.42 (s, 1H), 5.29 (d, J = 1.2 Hz, 1H), 1.67 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.9, 139.0, 137.0, 136.9, 131.9, 130.8, 129.7, 129.4, 129.3, 129.0, 125.1, 19.1; ESI-HRMS (ESI, m/z): Calcd for $C_{16}H_{14}BrNO_3SNa$, $[M + Na]^+$: 401.9775, found 401.9778.

4-Bromo-N-(3-oxo-3-phenylpropyl)-N-phenylbenzenesulfonamide (8)



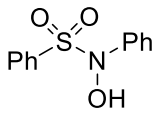
The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 20 : 1), White solid; (83 mg, 94%); mp: 145–146 °C; R_f = 0.50 (hexanes/ethyl acetate 5: 1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87–7.85 (m, 2H), 7.62–7.59 (m, 2H), 7.57–7.53 (m, 1H), 7.48–7.41 (m, 4H), 7.36–7.30 (m, 3H), 7.09–7.07 (m, 2H), 3.99 (t, J = 7.2 Hz, 2H), 3.28 (t, J = 7.2 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 197.7, 139.0, 136.8, 136.3, 133.4, 132.1, 129.3, 129.2, 128.7, 128.6, 128.3, 127.9, 127.8, 47.0, 38.2; ESI-HRMS (ESI, m/z): Calcd for $C_{21}H_{18}BrNO_3SNa$, $[M + Na]^+$: 466.0088, found 466.0091.

4'-Methoxy-N-phenyl-[1,1'-biphenyl]-4-sulfonamide (9)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 20 : 1), White solid; (49 mg, 72%); mp: 157–159 °C; R_f = 0.35 (hexanes/ethyl acetate 10: 1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 8.4 Hz, 2H), 7.57 (d, J = 8.4 Hz, 2H), 7.49–7.47 (m, 2H), 7.30 (brs, 1H), 7.25–7.22 (m, 2H), 7.15–7.08 (m, 3H), 6.98–6.95 (m, 2H), 3.84 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 160.0, 145.4, 136.7, 136.5, 131.4, 129.3, 128.4, 127.7, 126.9, 125.3, 121.4, 114.4, 55.3; ESI-HRMS (ESI, m/z): Calcd for $C_{19}H_{17}NO_3SNa$, $[M + Na]^+$: 362.0827, found 362.0828.

N-Hydroxy-N-phenylbenzenesulfonamide (12)



The product was obtained by silica gel column chromatography (hexane/ethyl acetate = 10 : 1), Yellow solid; (19 mg, 38%); mp: 108–109 °C; R_f = 0.30 (hexanes/ethyl acetate 5: 1);[13] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (t, J = 7.6 Hz, 1H), 7.53 (d, J = 7.6 Hz, 2H), 7.43 (t, J = 7.6 Hz, 2H), 7.26–7.24 (m, 3H), 7.15–7.13 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.4, 133.9, 131.8, 129.8, 128.4, 128.3, 127.5, 122.9; ESI-HRMS (ESI, m/z): Calcd for $C_{12}H_{12}NO_3S$, $[M + H]^+$: 250.0538, found 250.0533.

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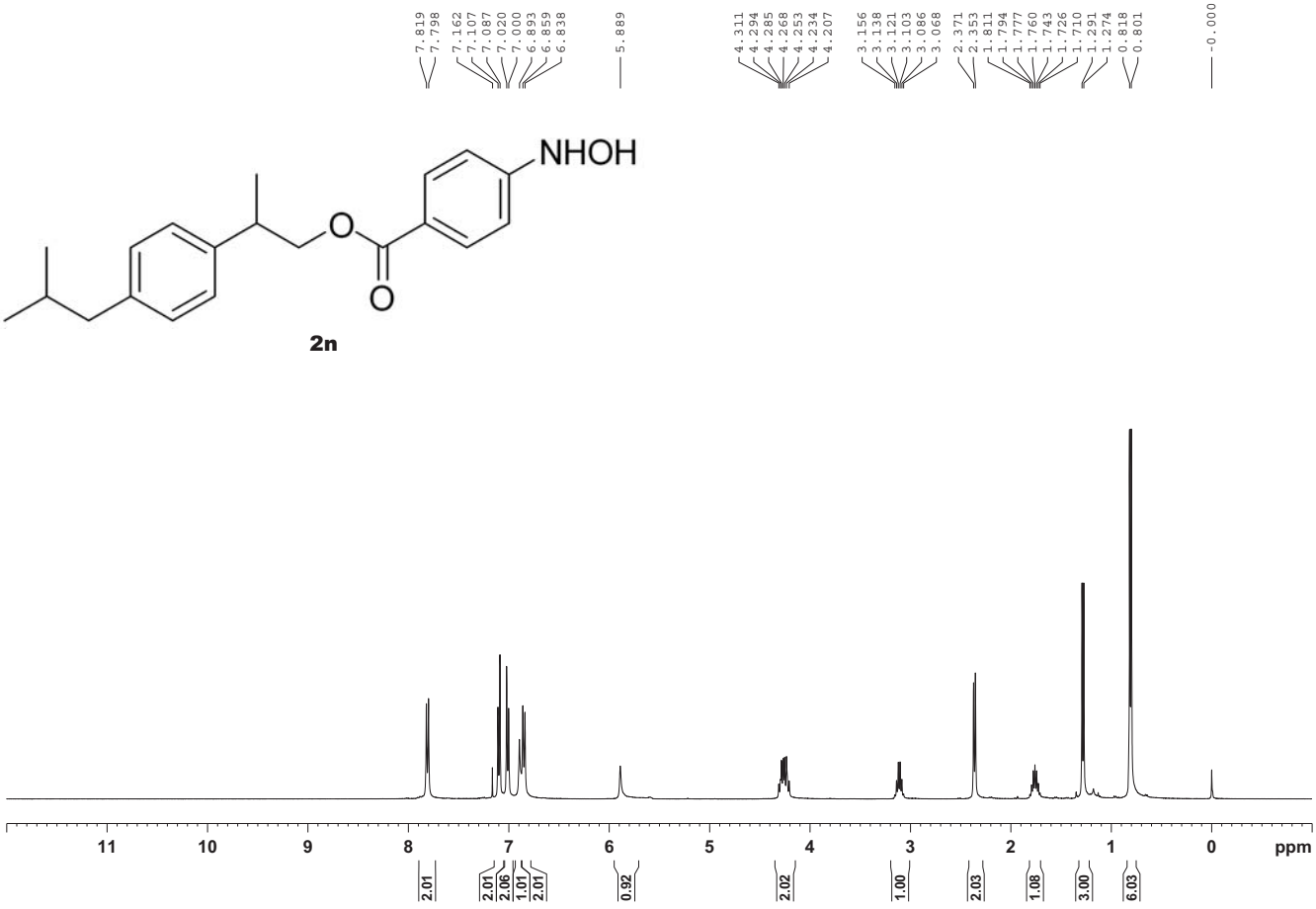
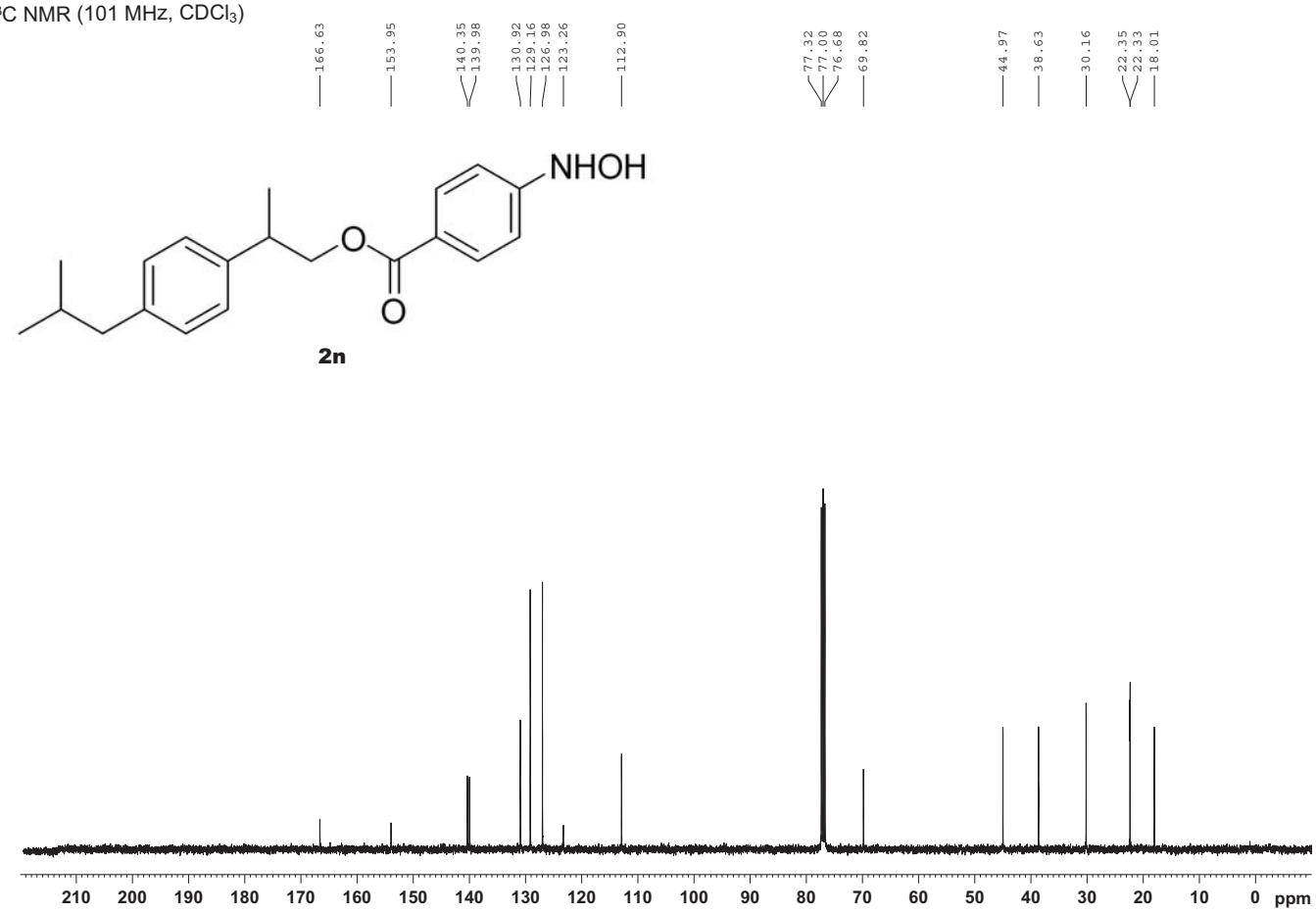
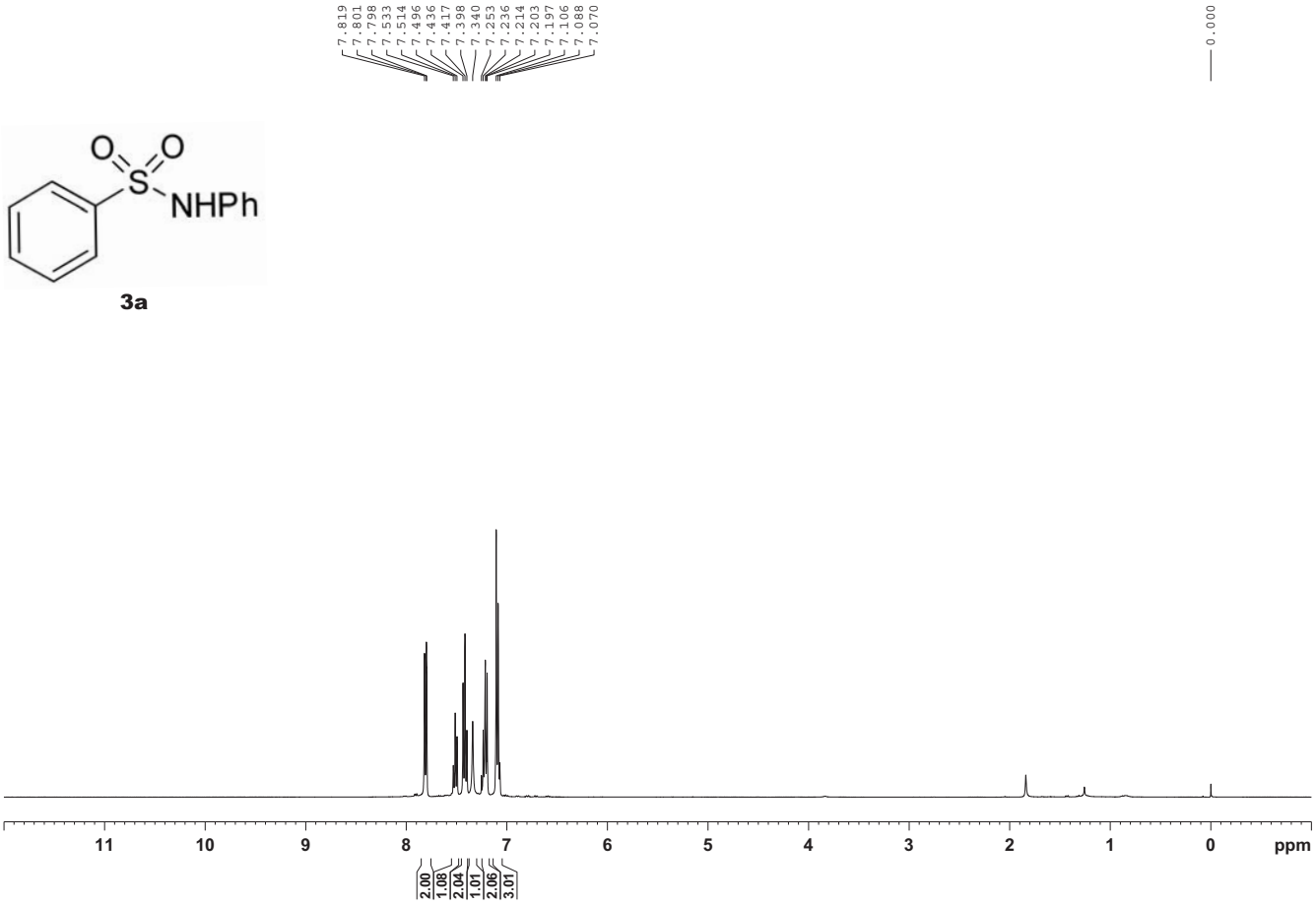
^{1}H NMR (400 MHz, $CDCl_3$)



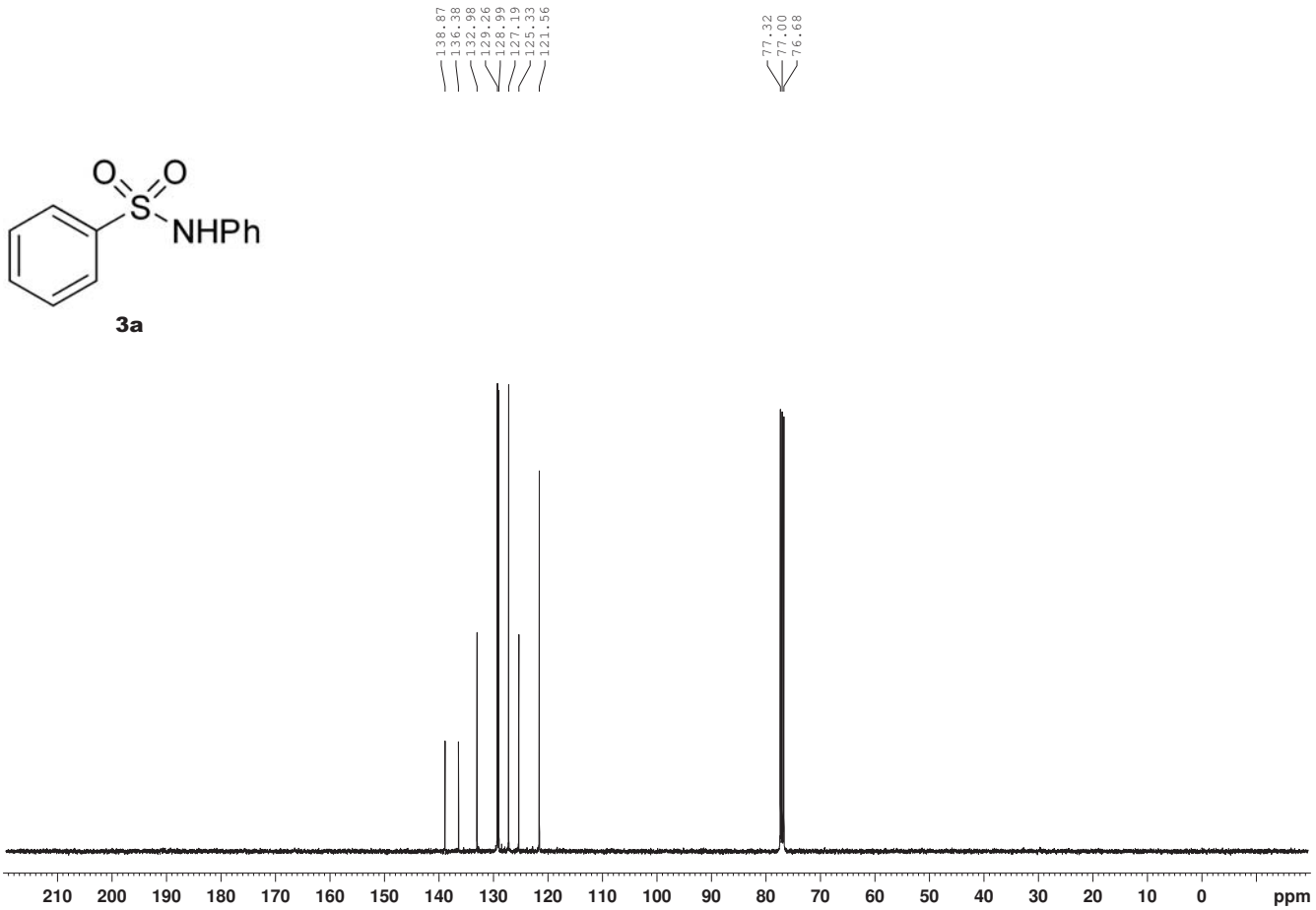
^{13}C NMR (101 MHz, $CDCl_3$)



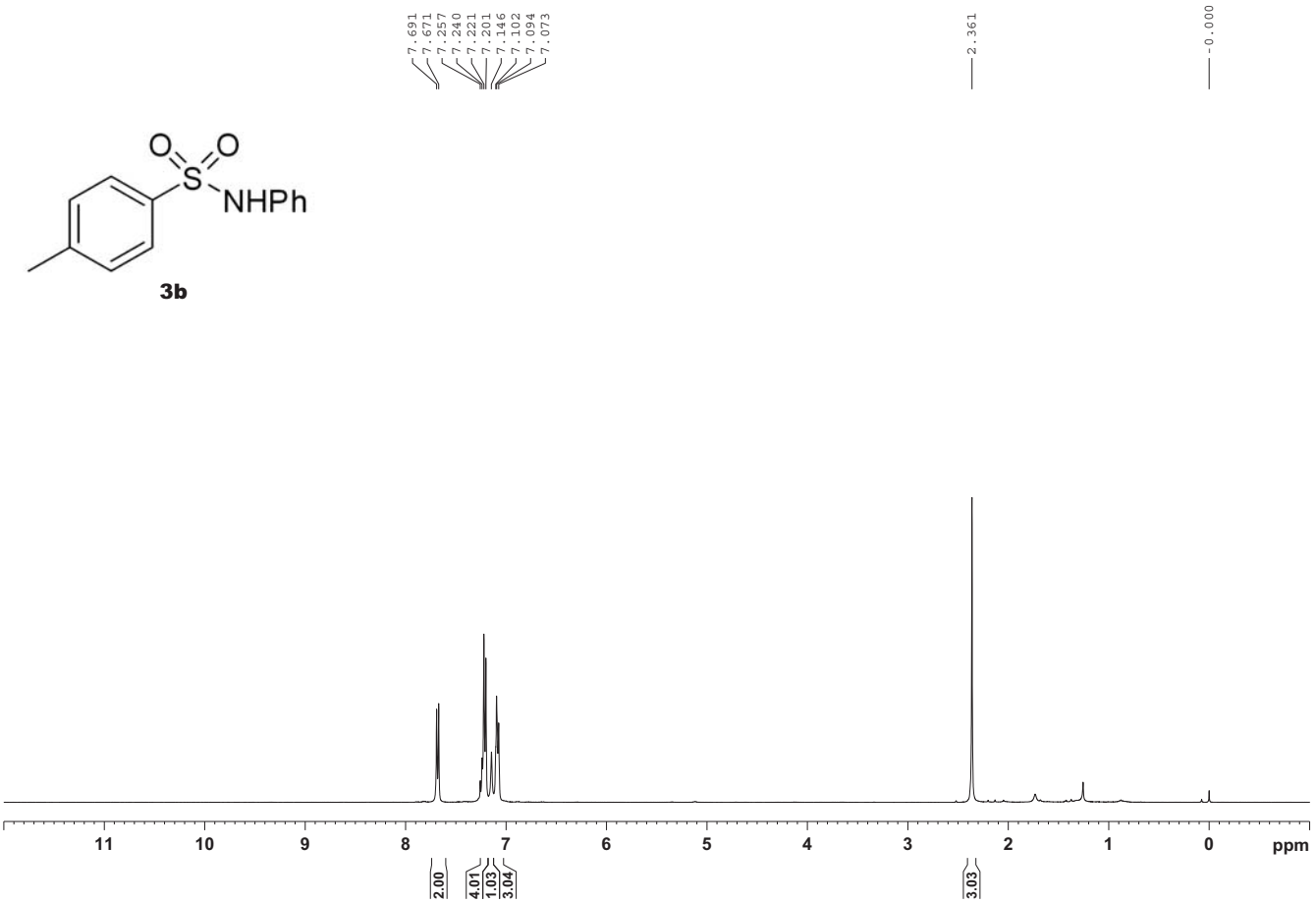
^{1}H NMR (400 MHz, $CDCl_3$)



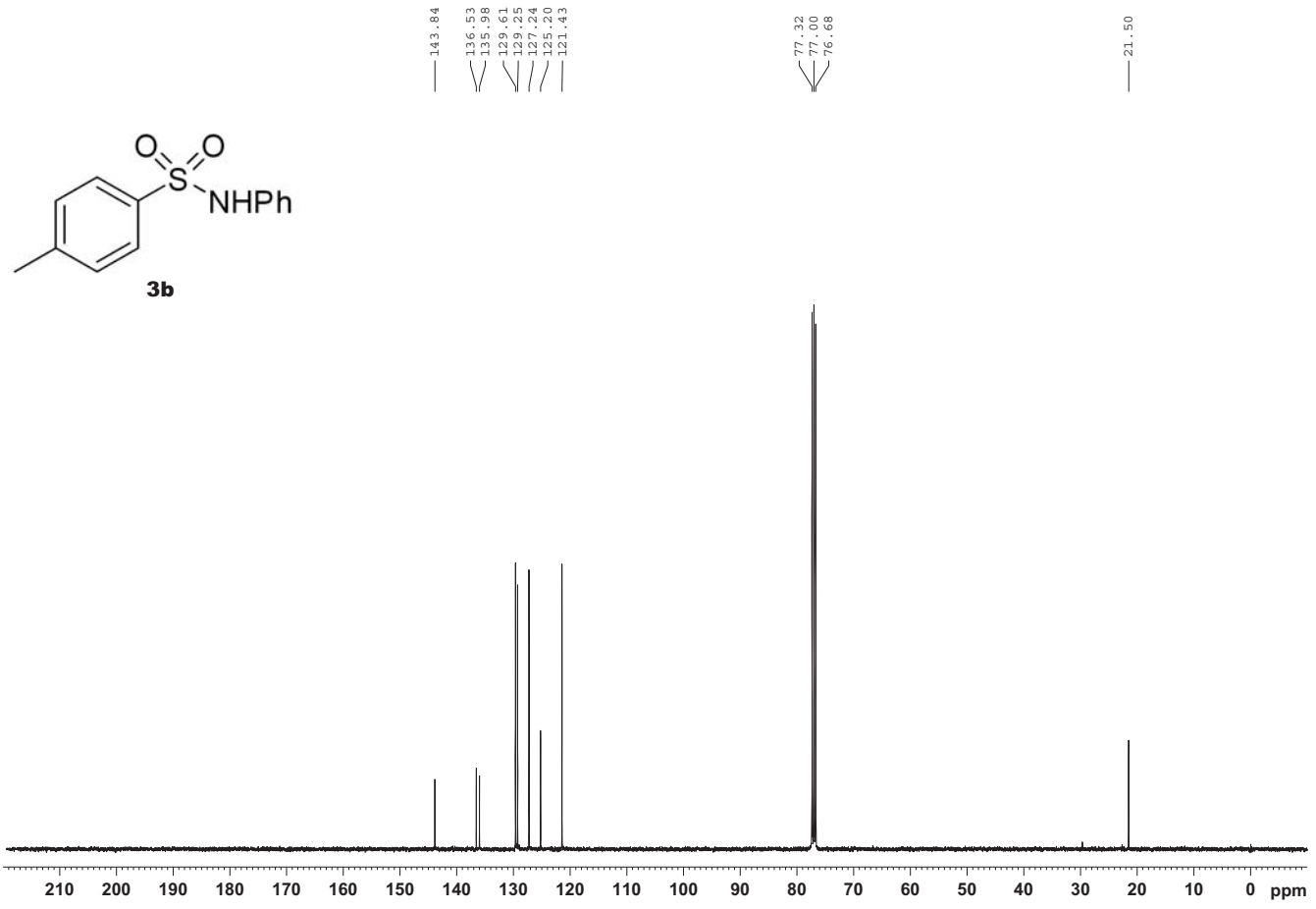
^{13}C NMR (101 MHz, $CDCl_3$)



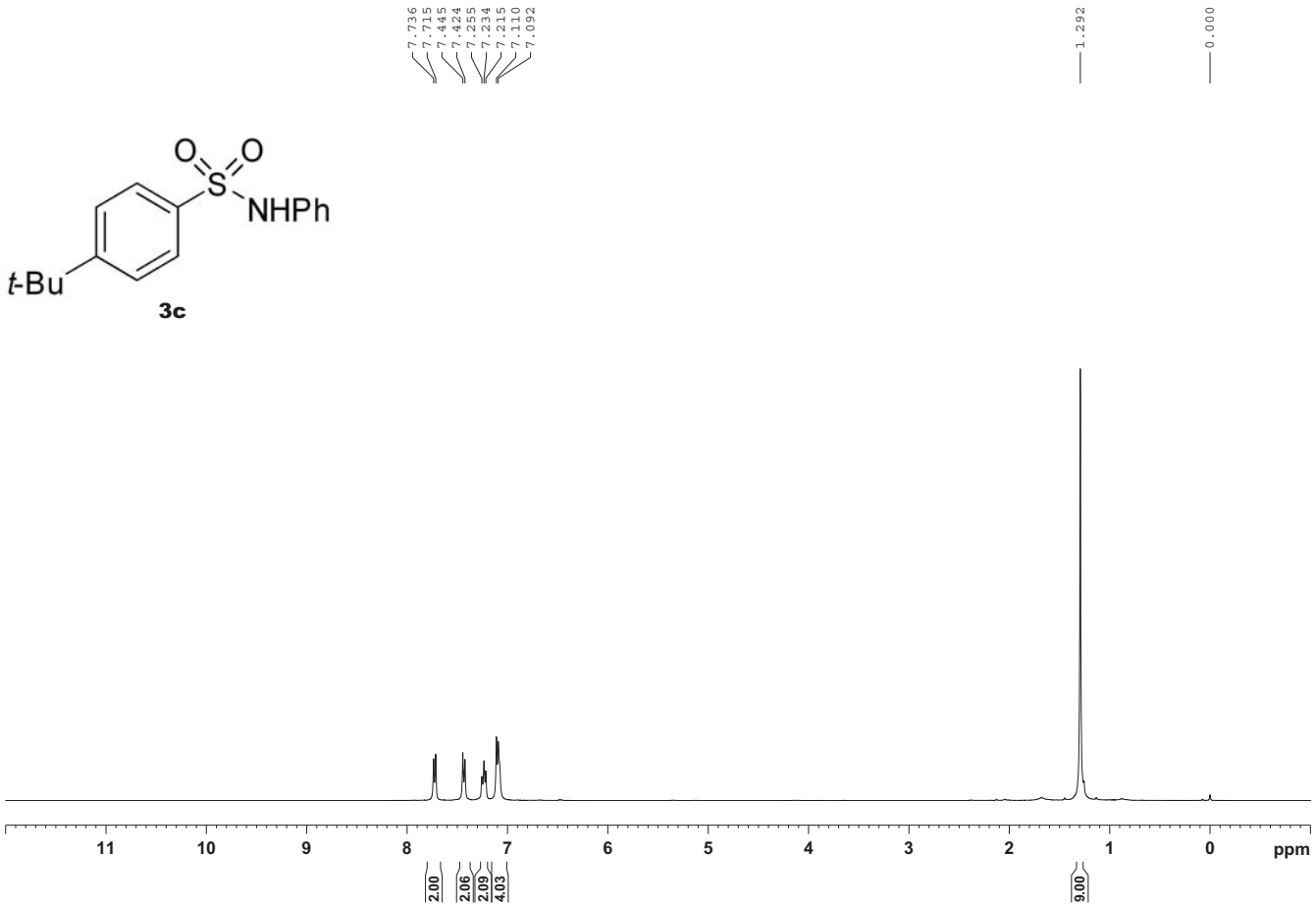
^{1}H NMR (400 MHz, $CDCl_3$)



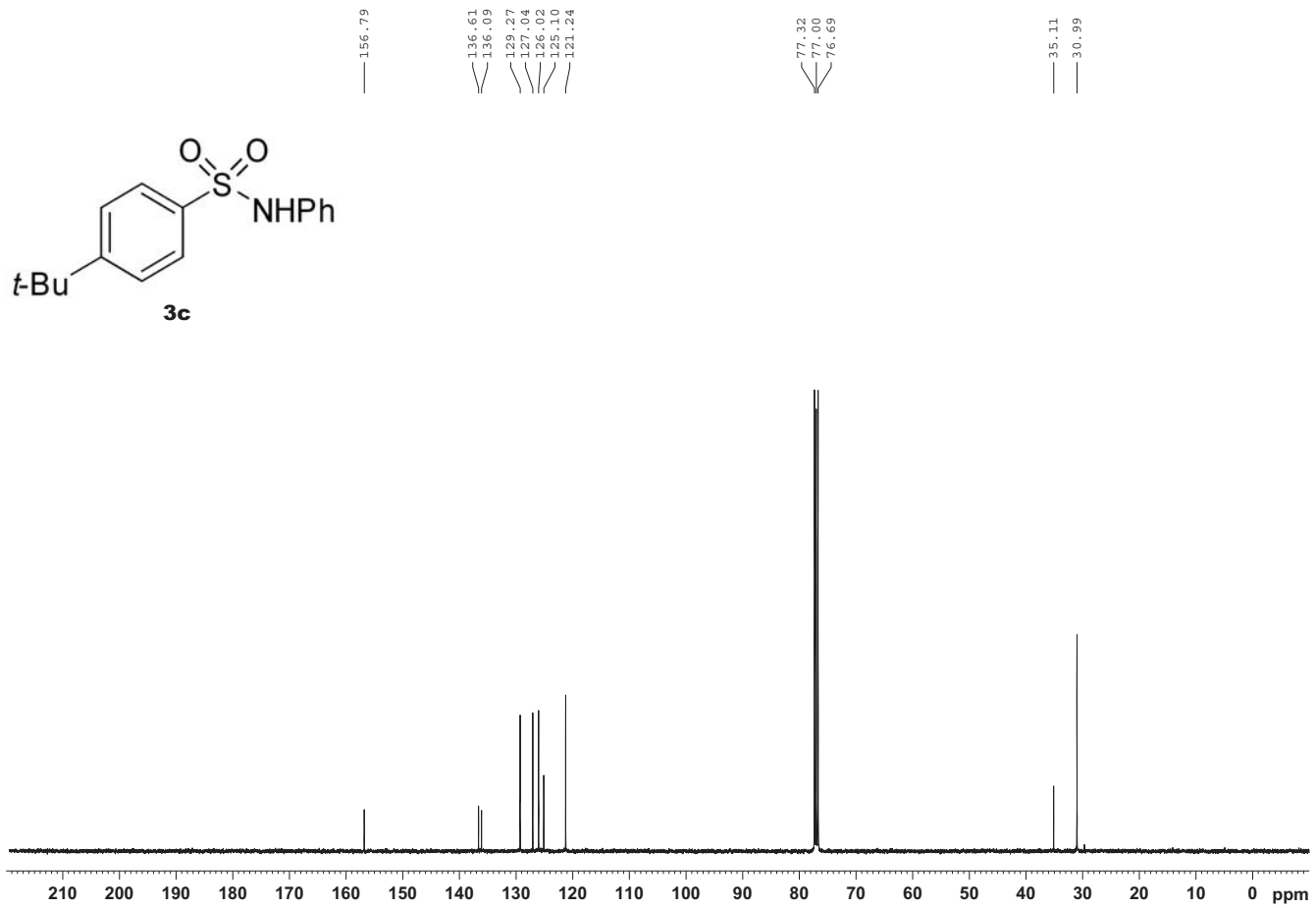
^{13}C NMR (101 MHz, $CDCl_3$)



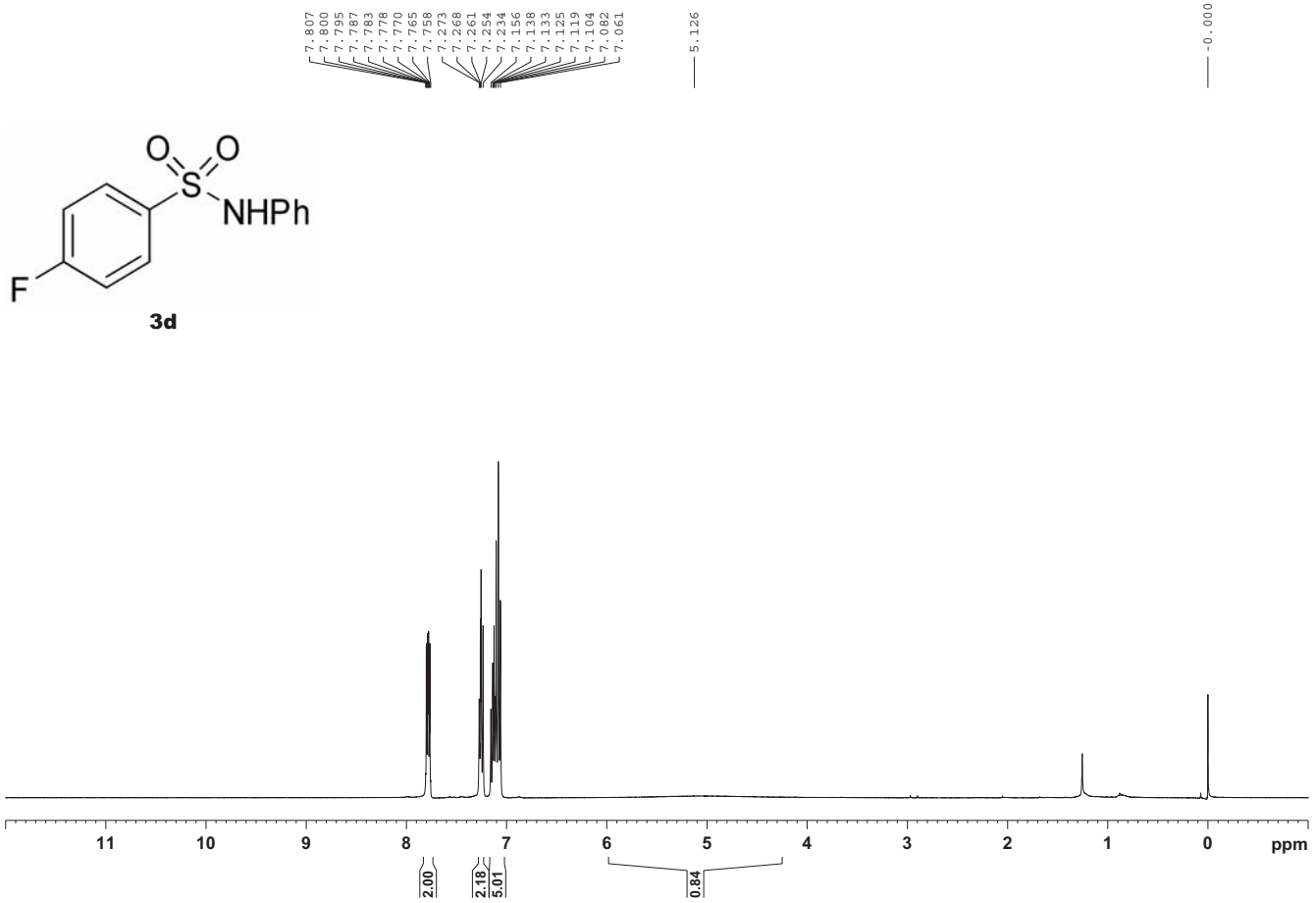
^{1}H NMR (400 MHz, $CDCl_3$)



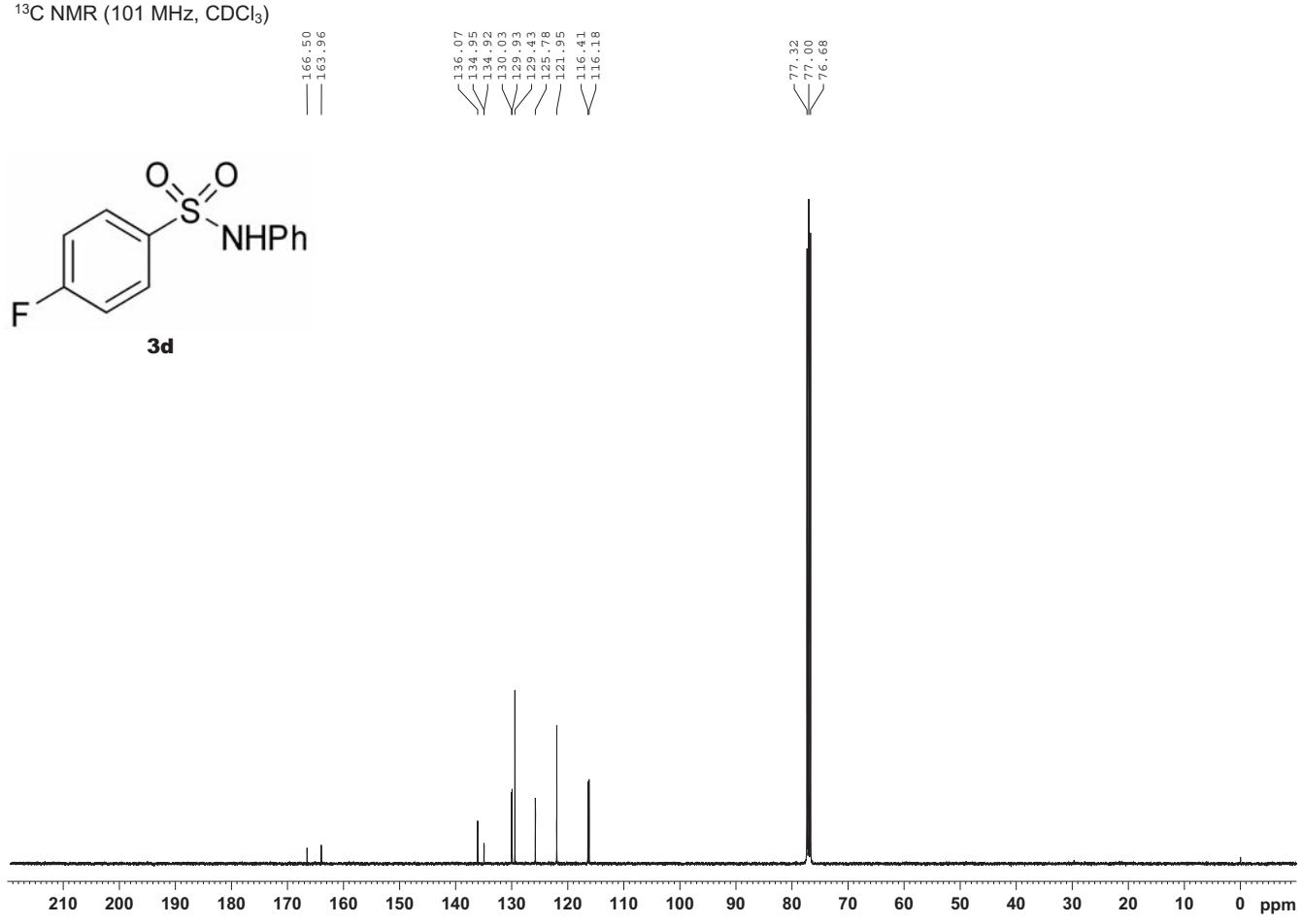
^{13}C NMR (101 MHz, $CDCl_3$)



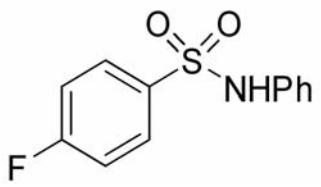
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)

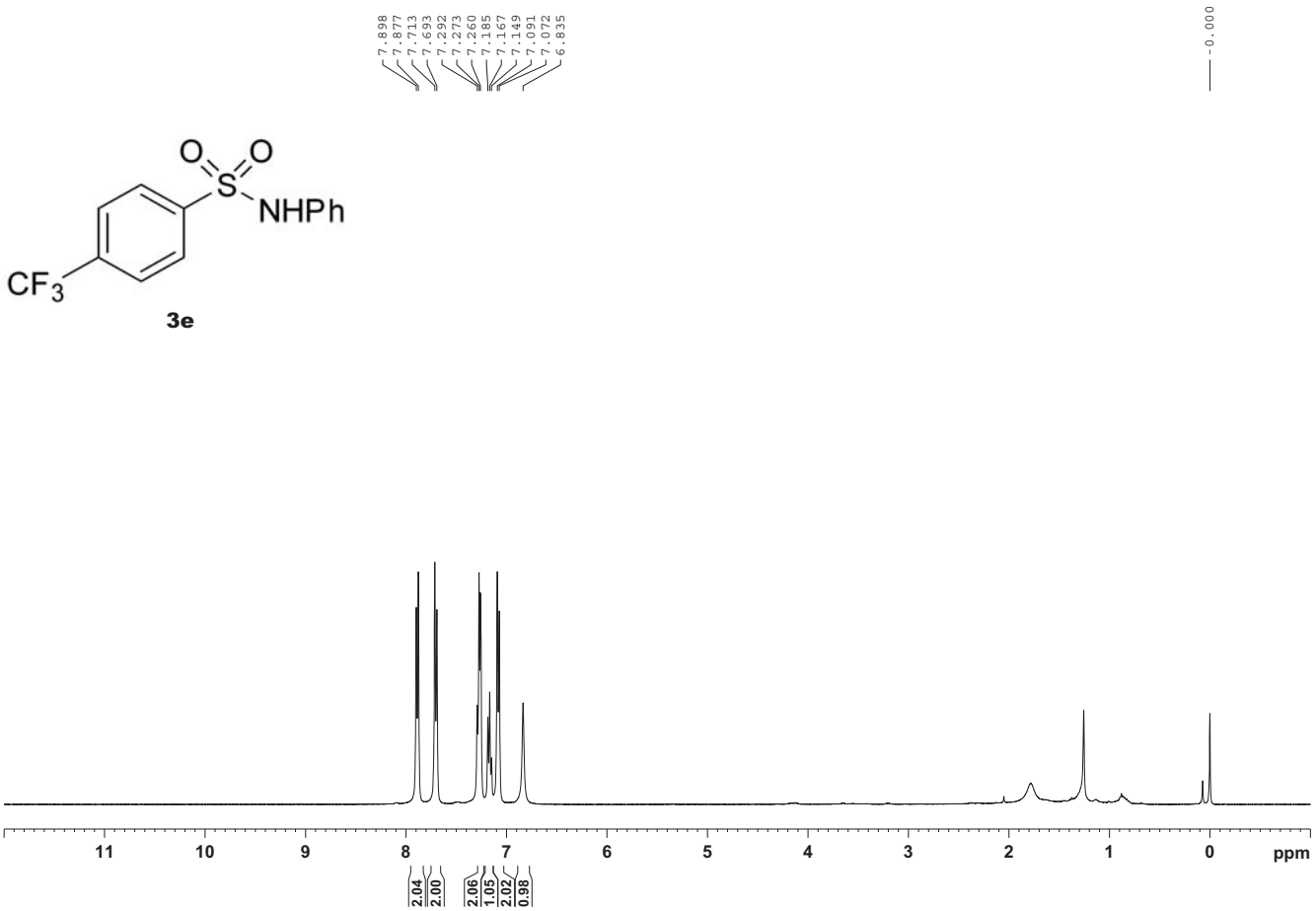


^{19}F NMR (376 MHz, $CDCl_3$)



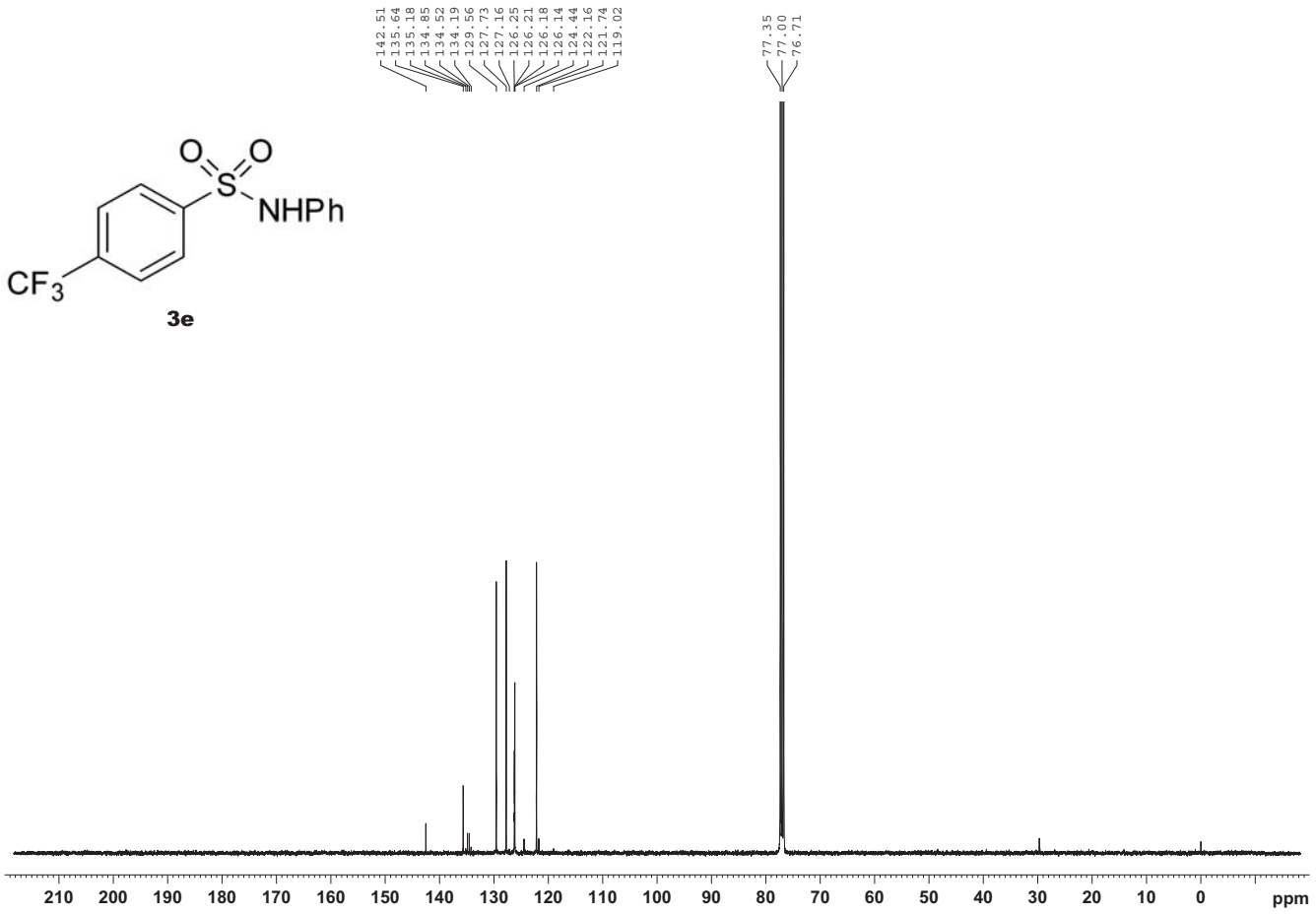
3d

^{1}H NMR (400 MHz, $CDCl_3$)

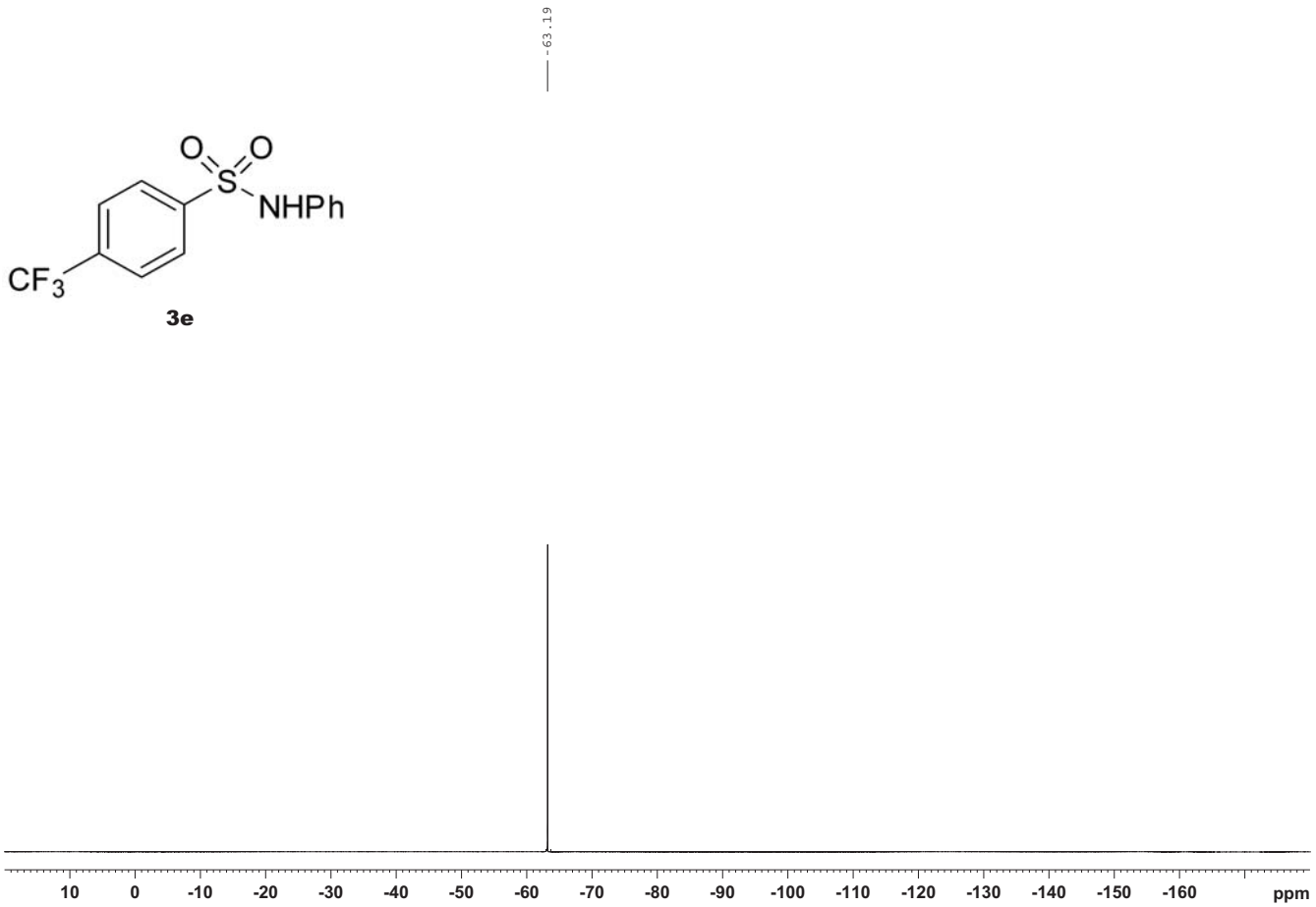


3e

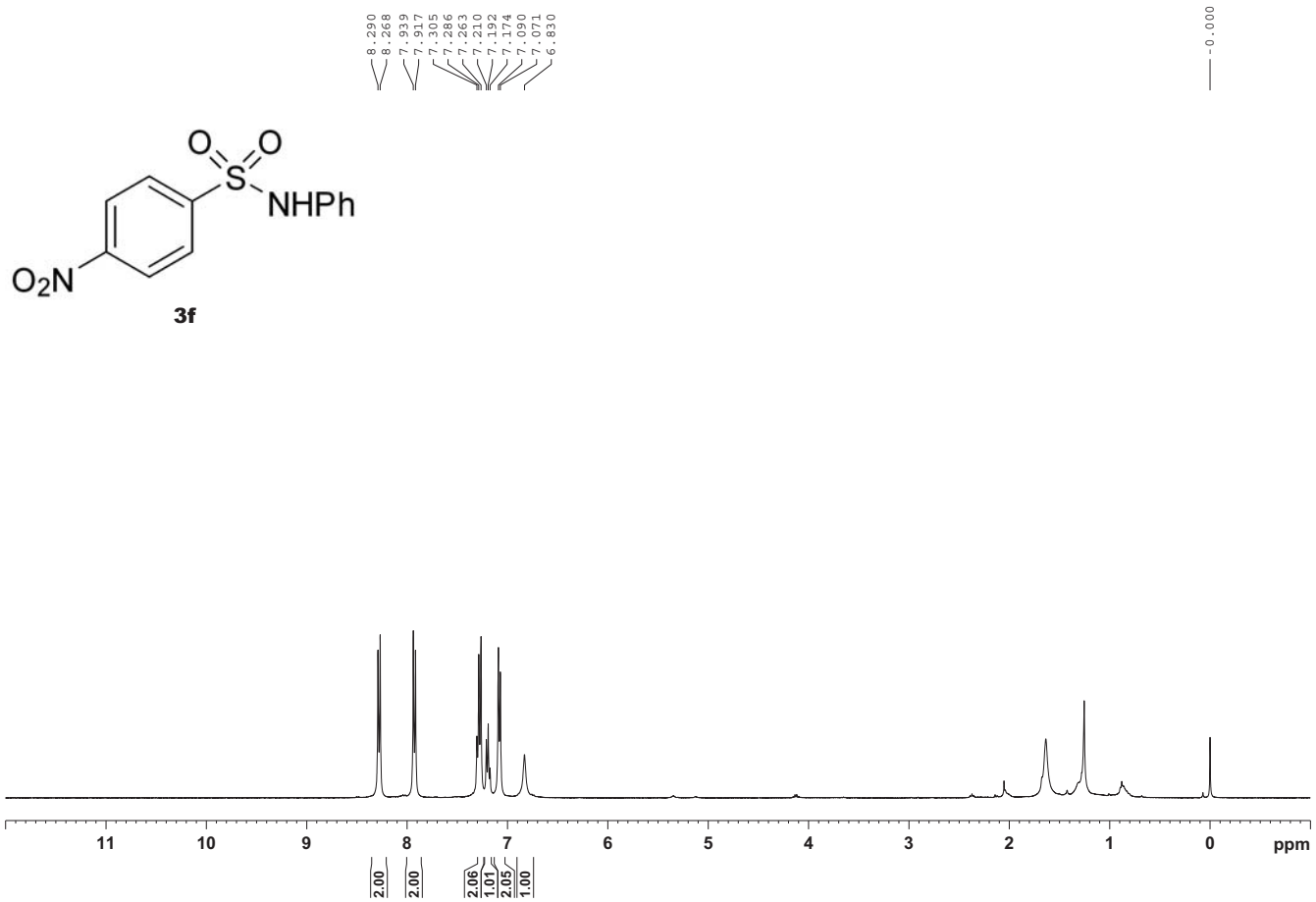
^{13}C NMR (101 MHz, $CDCl_3$)



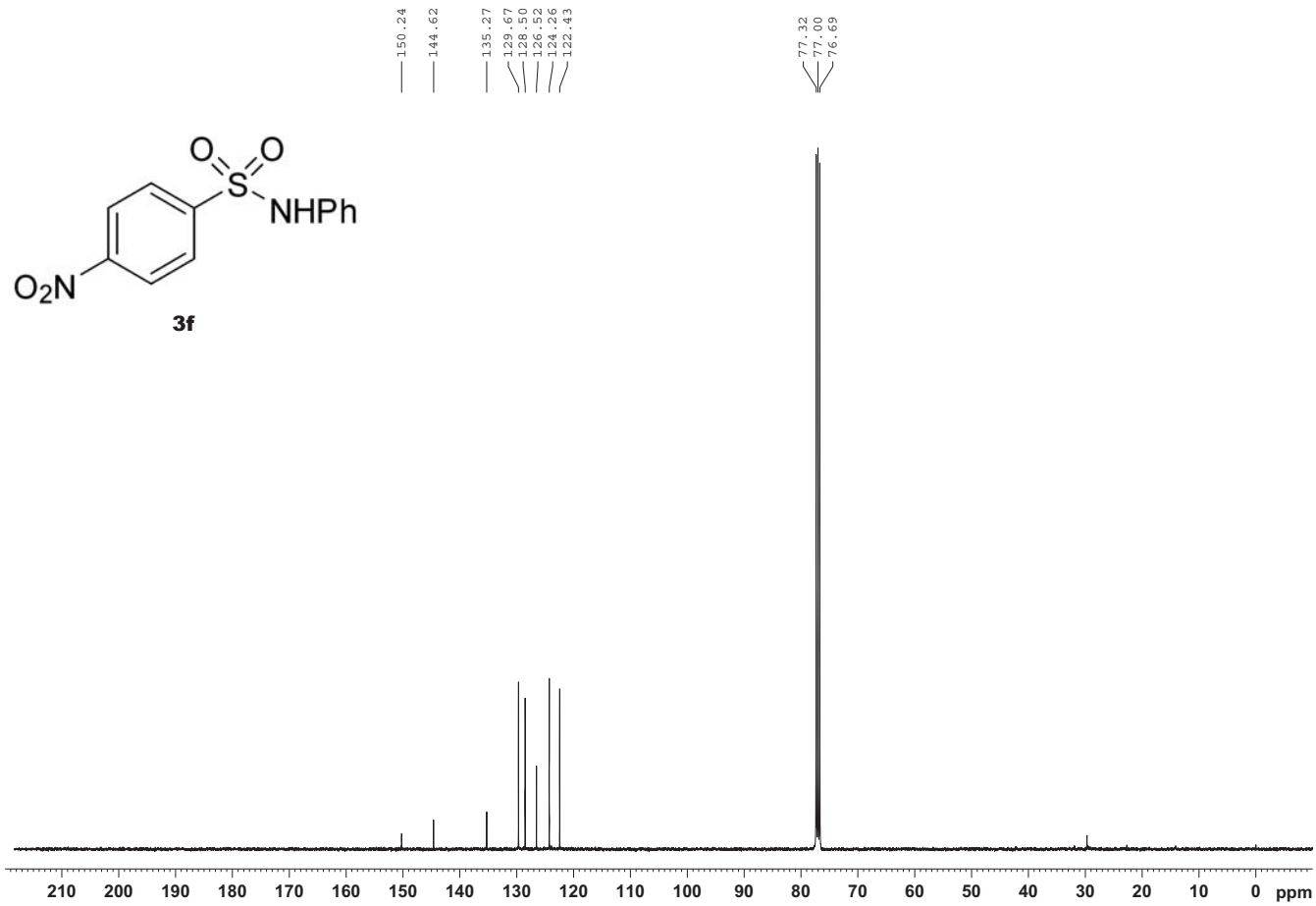
^{19}F NMR (376 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)



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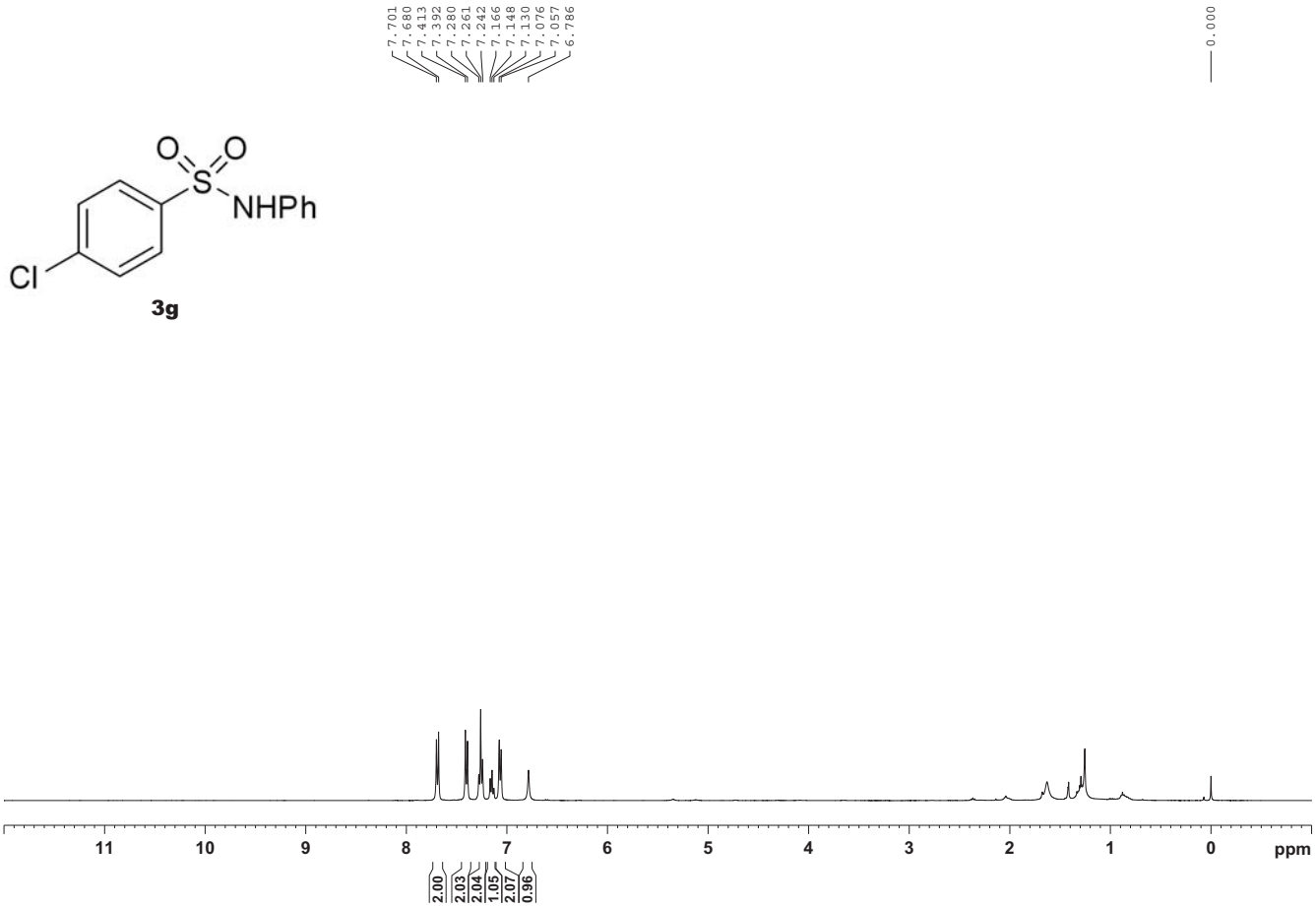
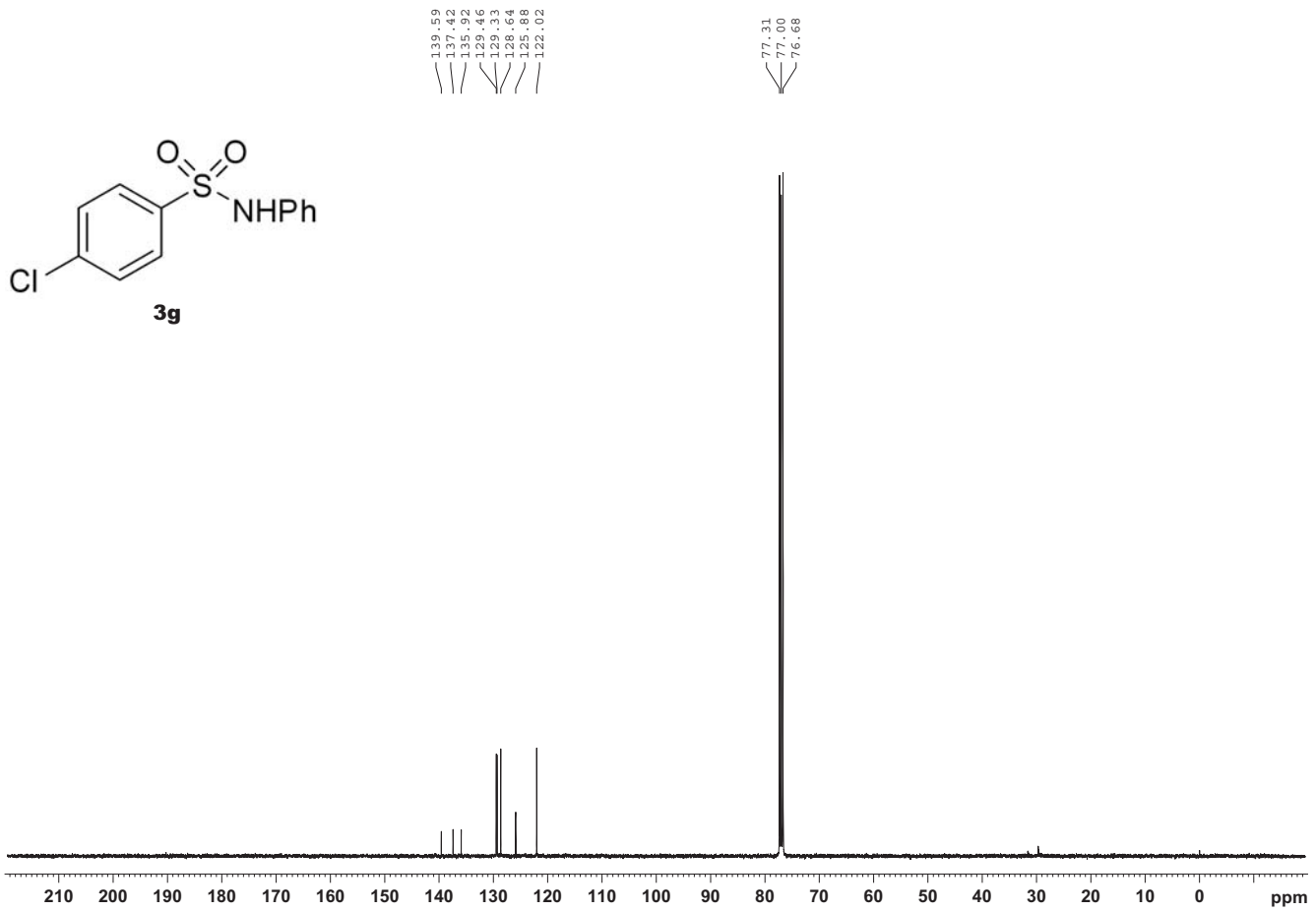
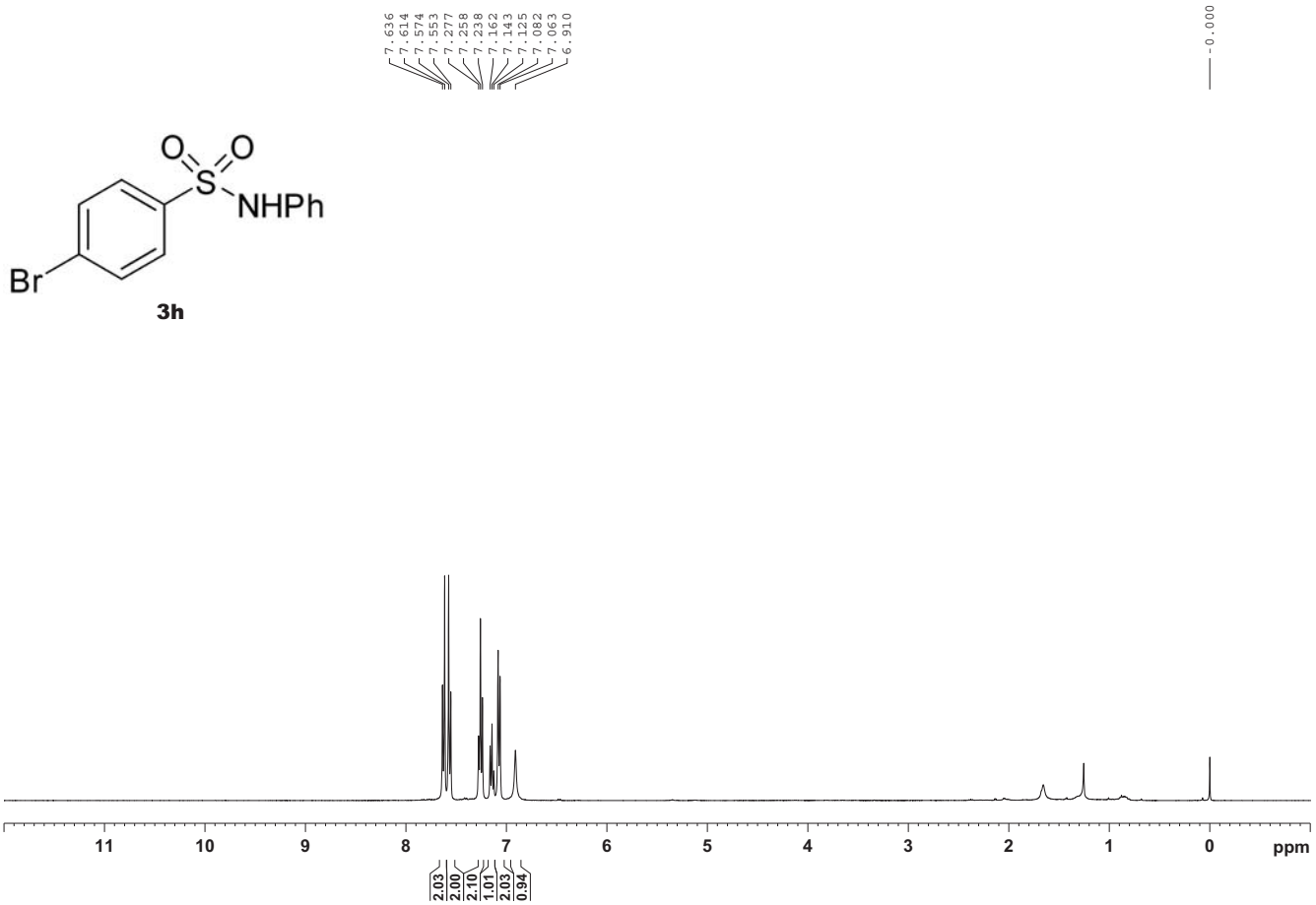


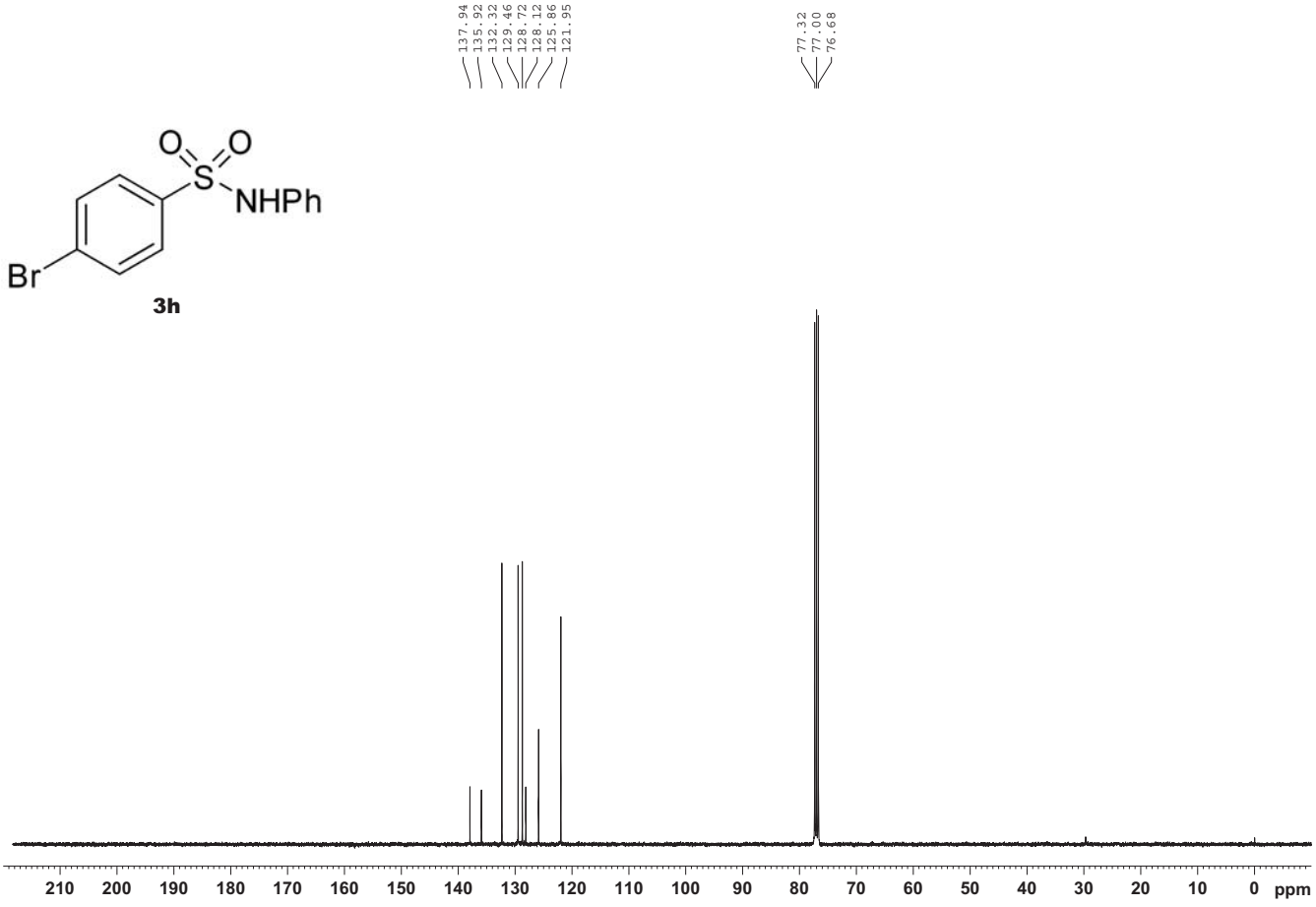
^{13}C NMR (101 MHz, $CDCl_3$)



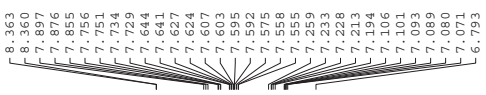
^{1}H NMR (400 MHz, $CDCl_3$)

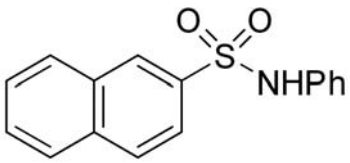


^{13}C NMR (101 MHz, $CDCl_3$)

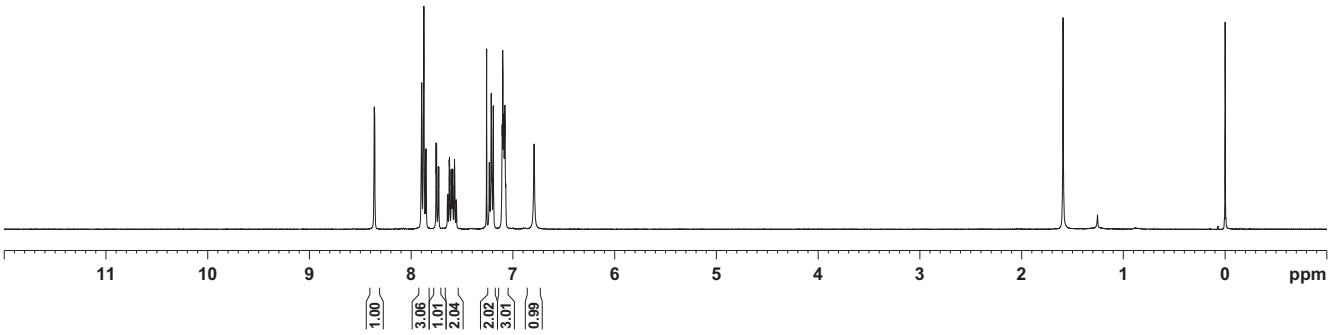


^{1}H NMR (400 MHz, $CDCl_3$)

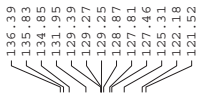


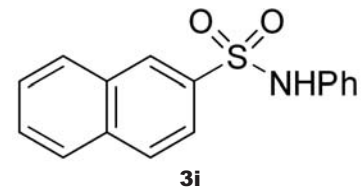


3i



^{13}C NMR (101 MHz, $CDCl_3$)





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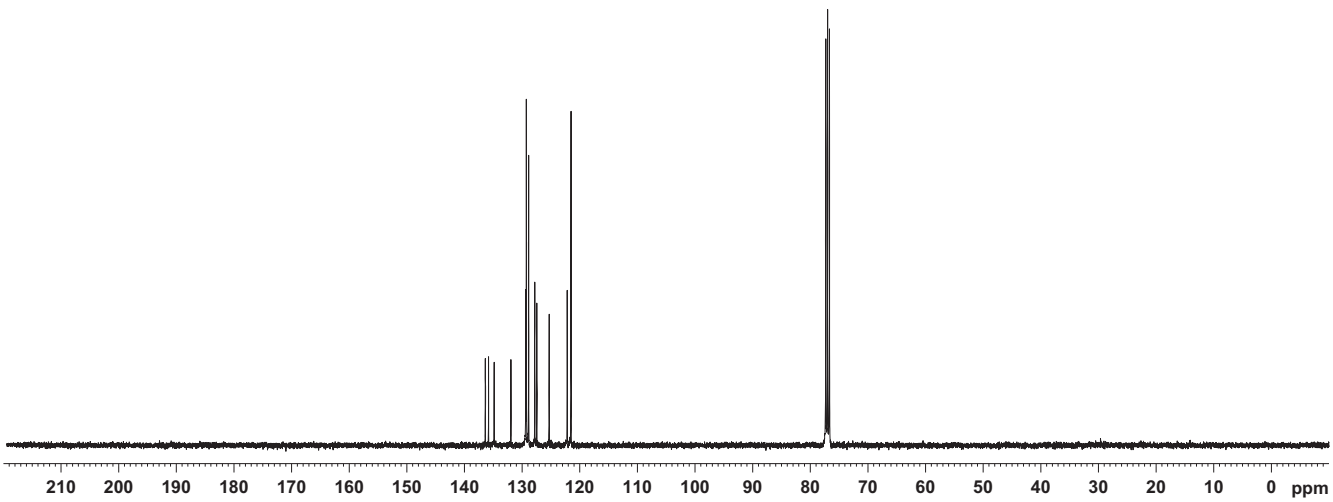
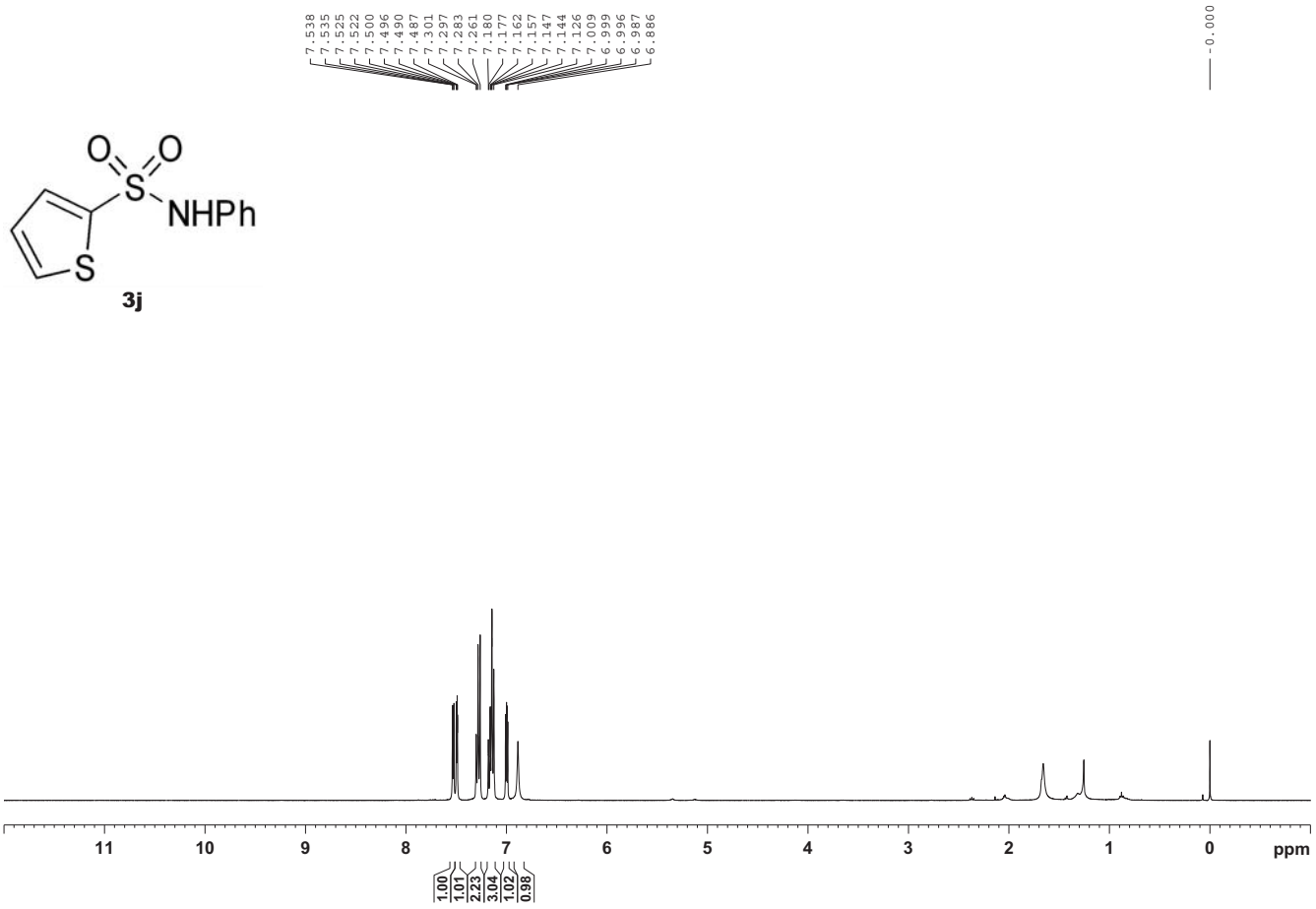
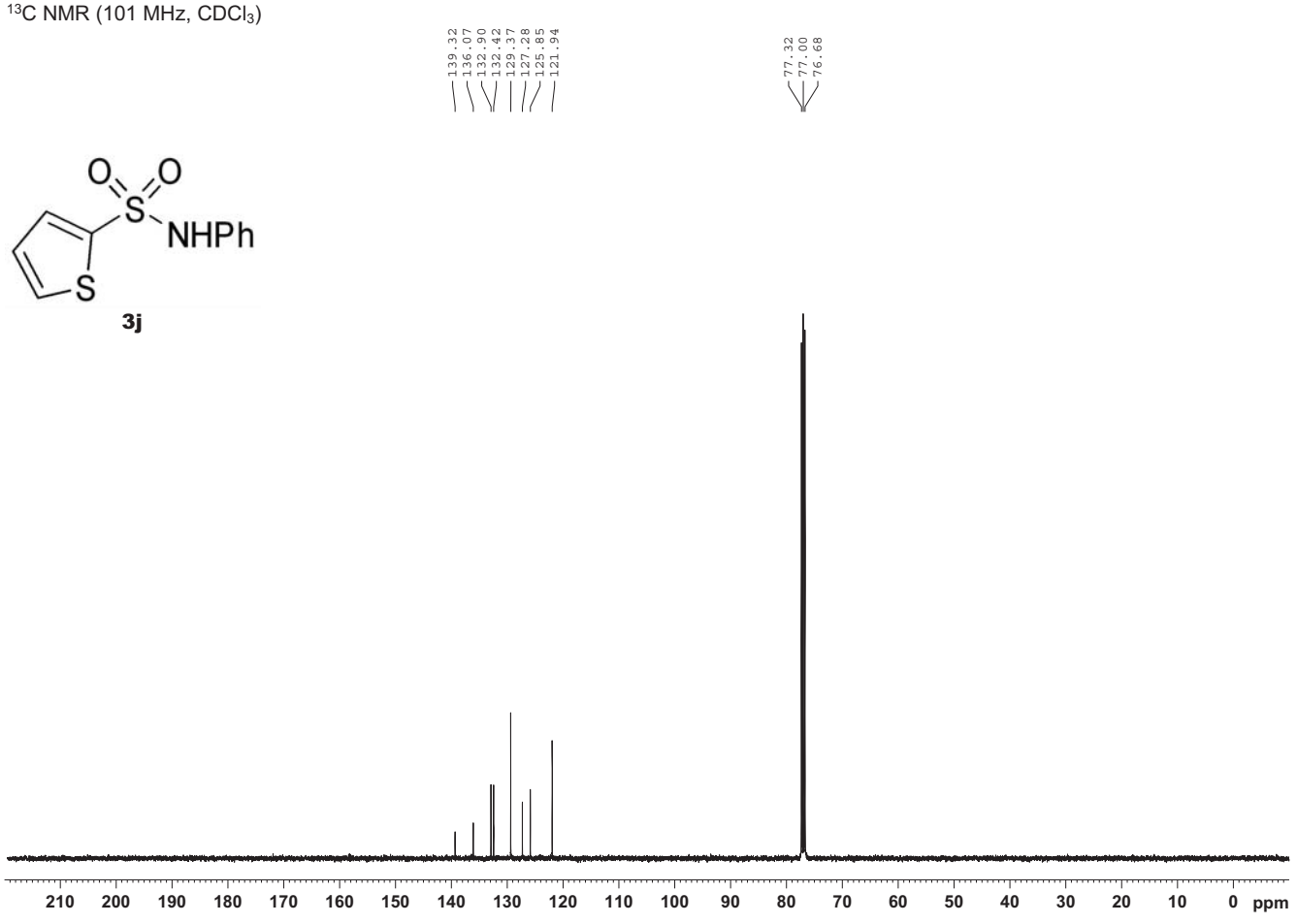


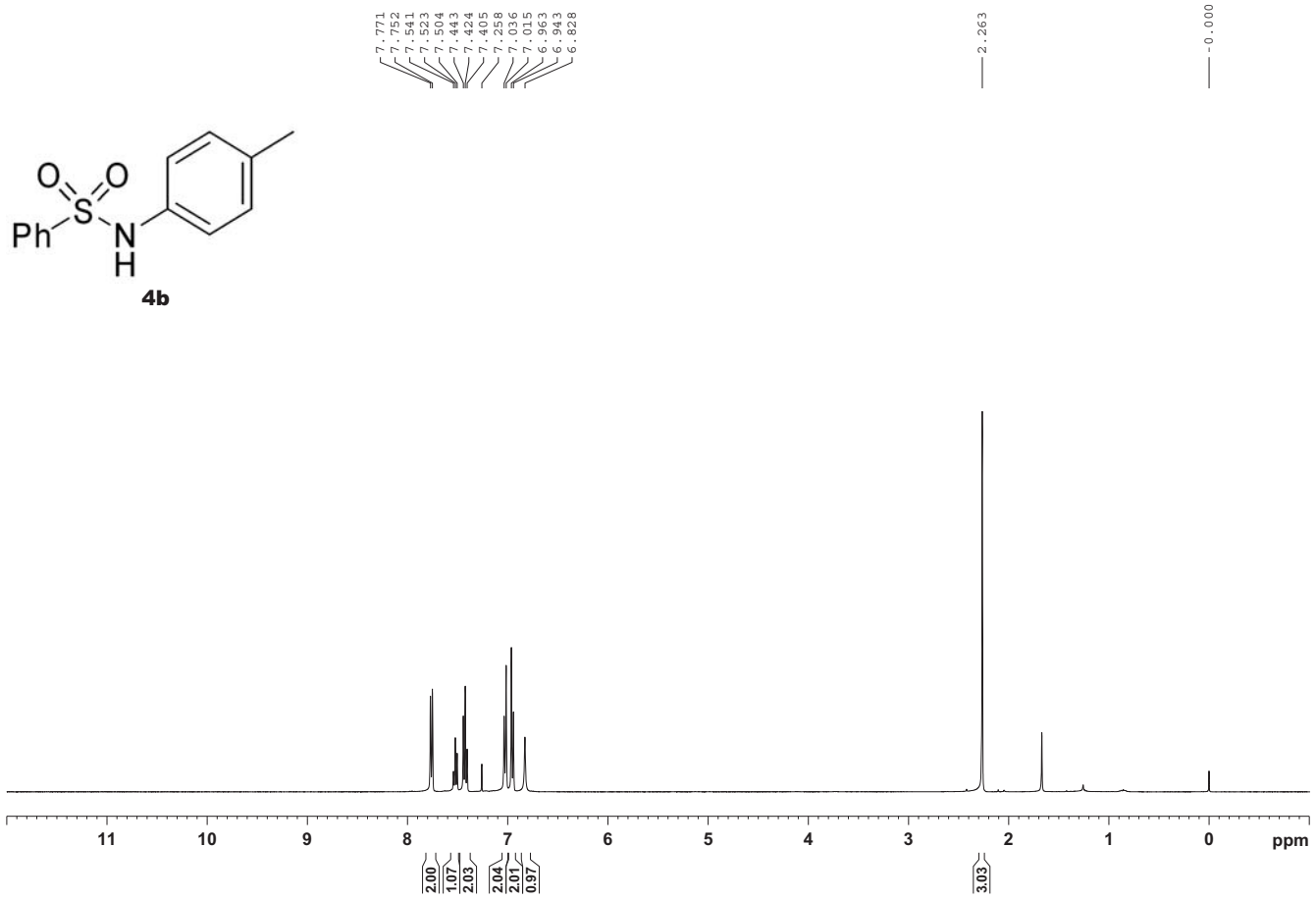
^{1}H NMR (400 MHz, $CDCl_3$)



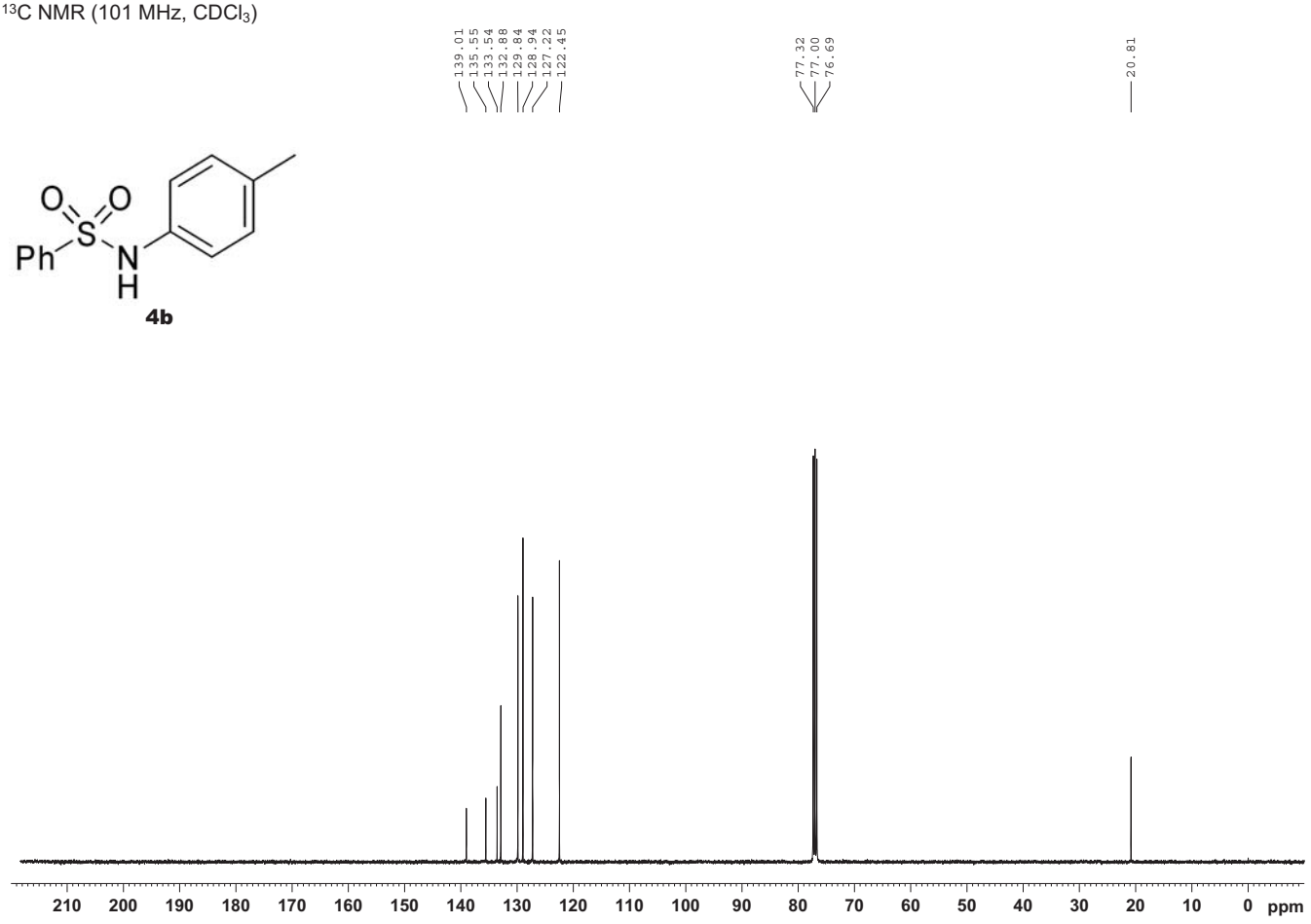
^{13}C NMR (101 MHz, $CDCl_3$)



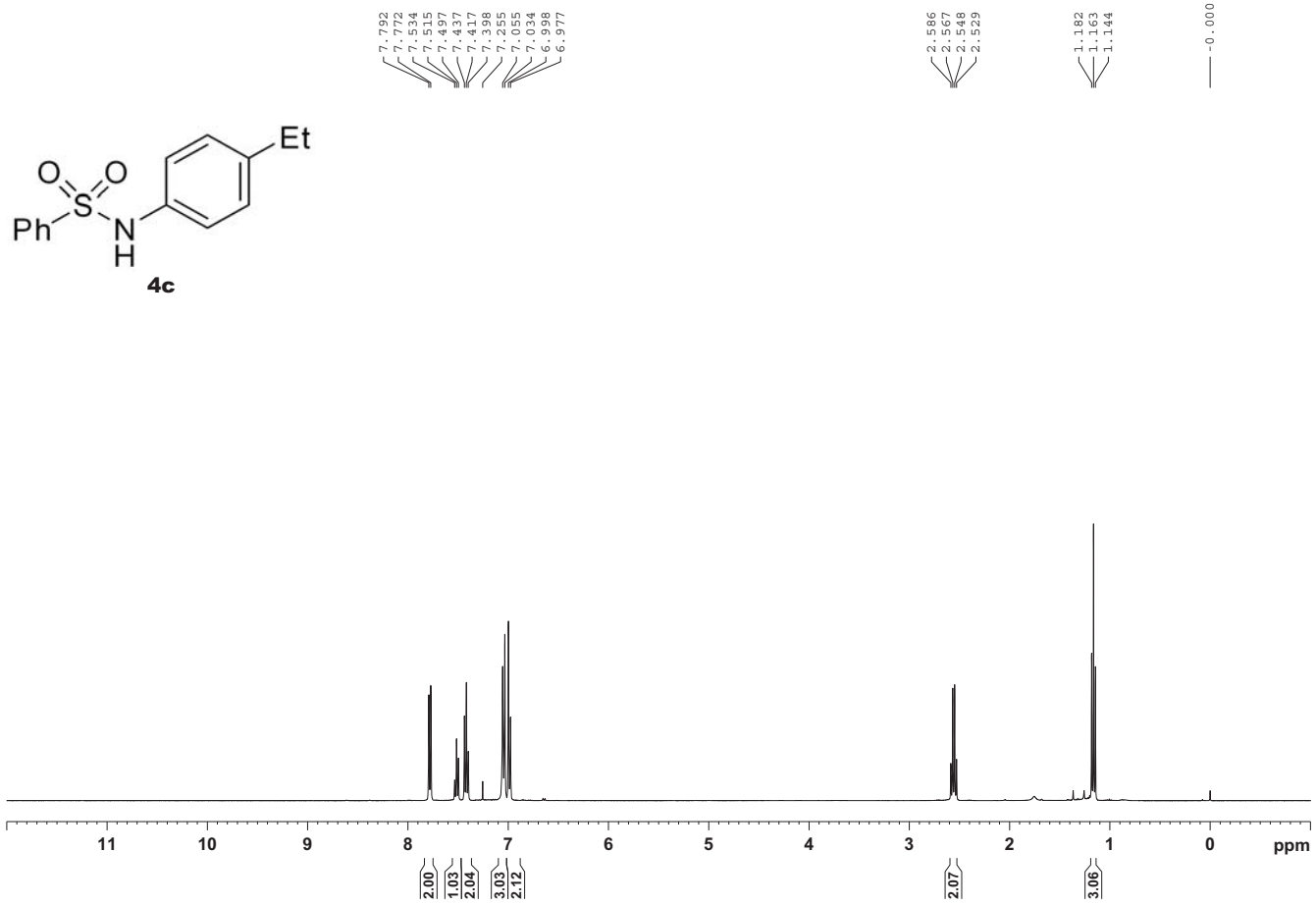
^{1}H NMR (400 MHz, $CDCl_3$)



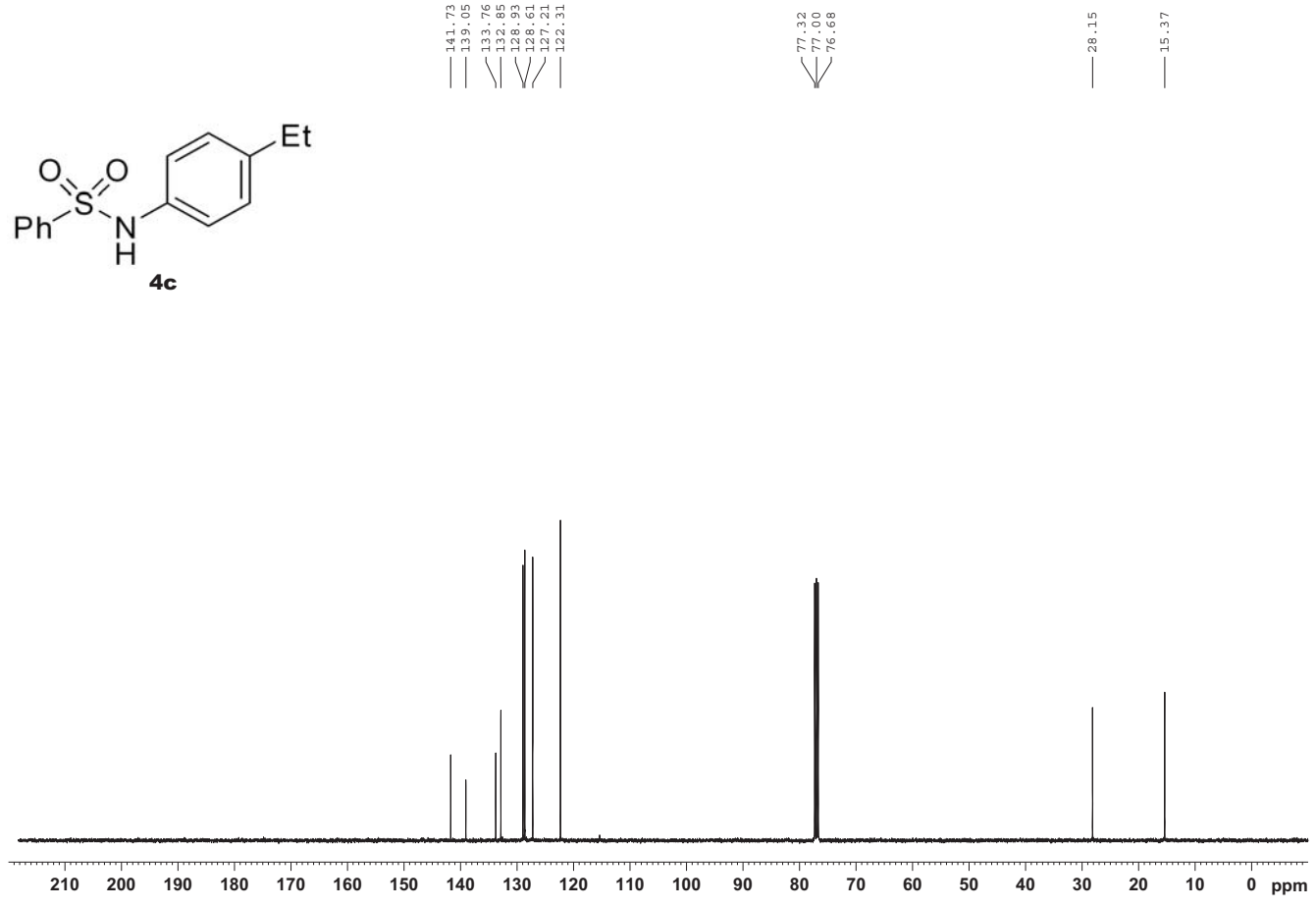
^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)



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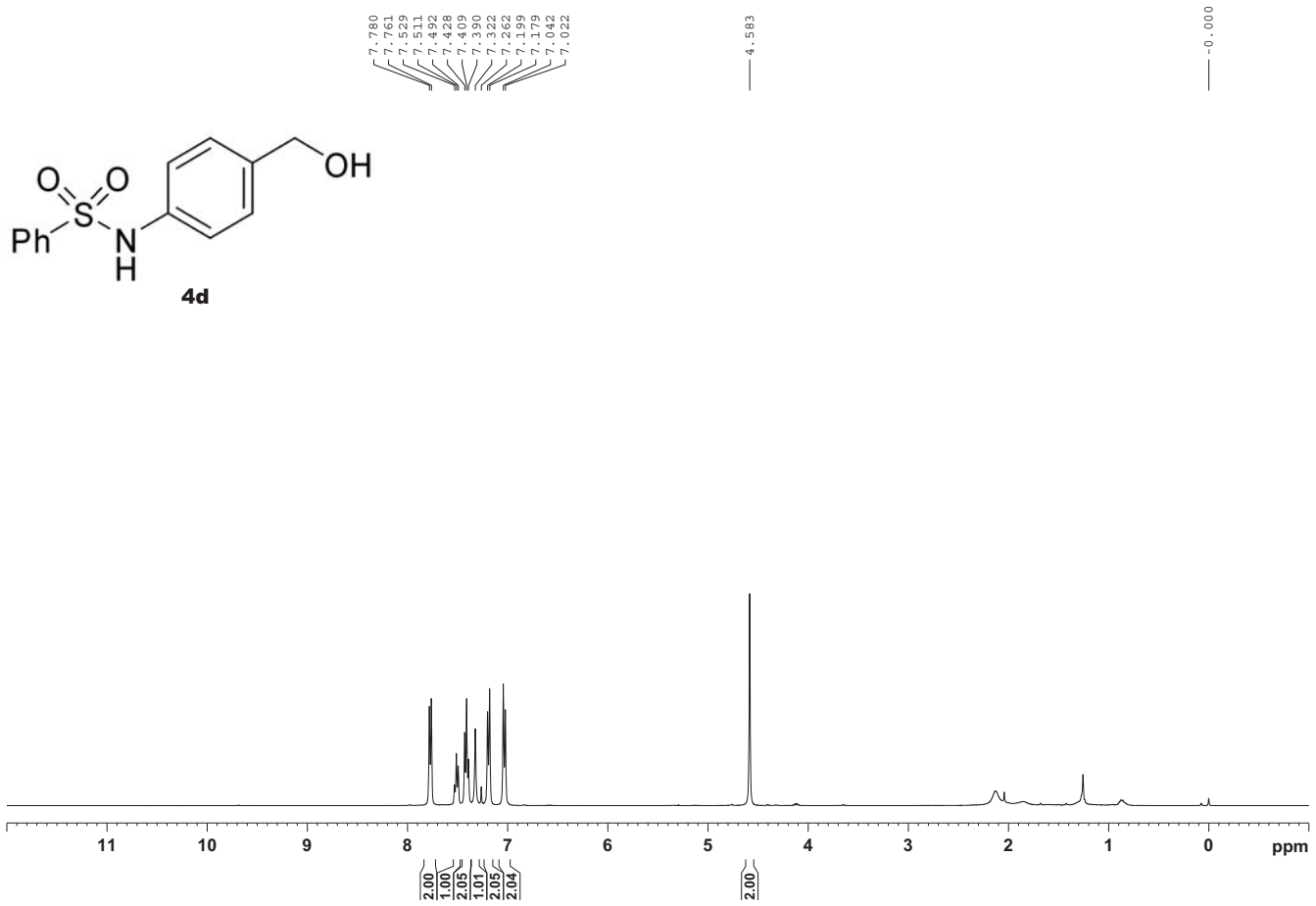
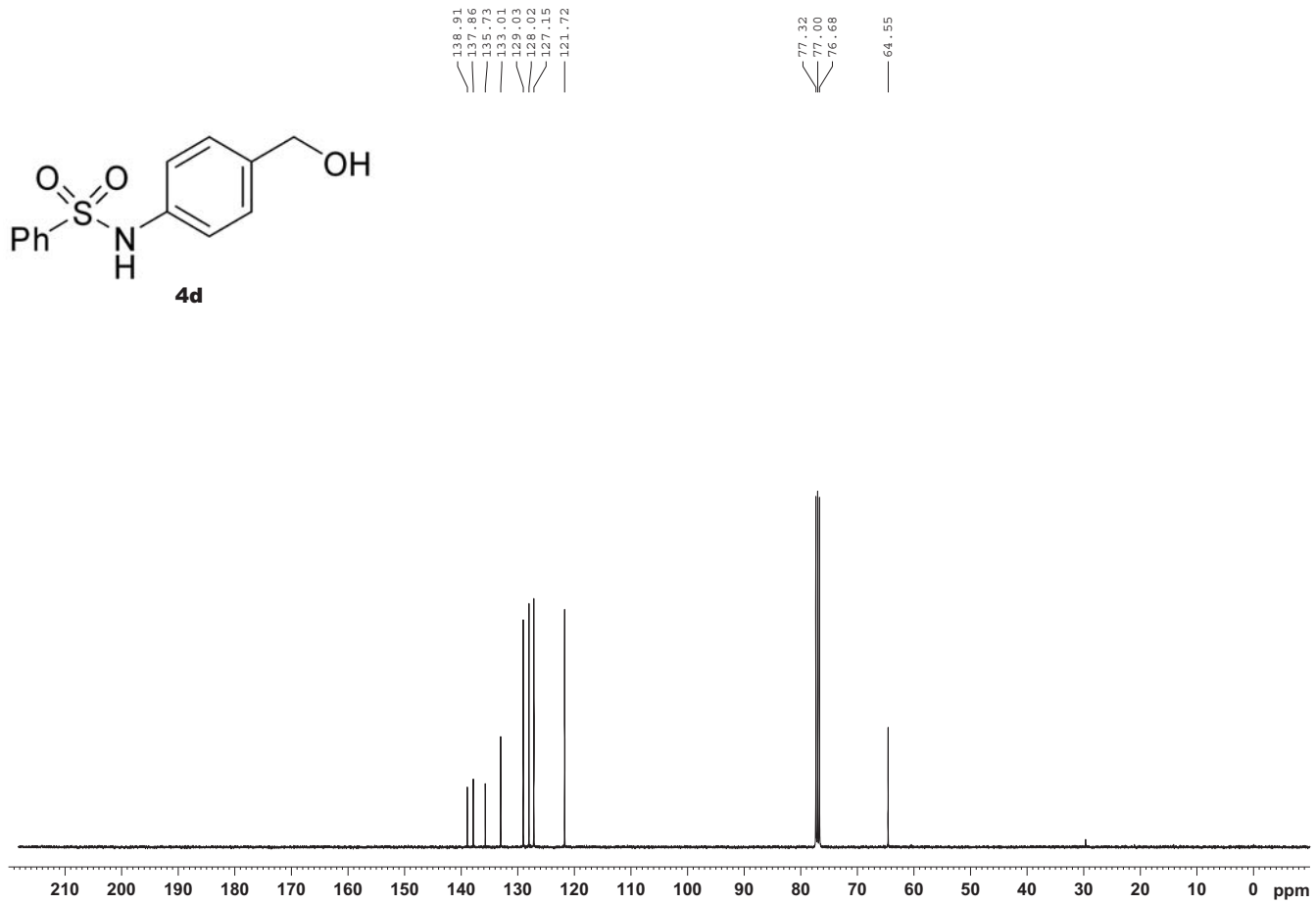
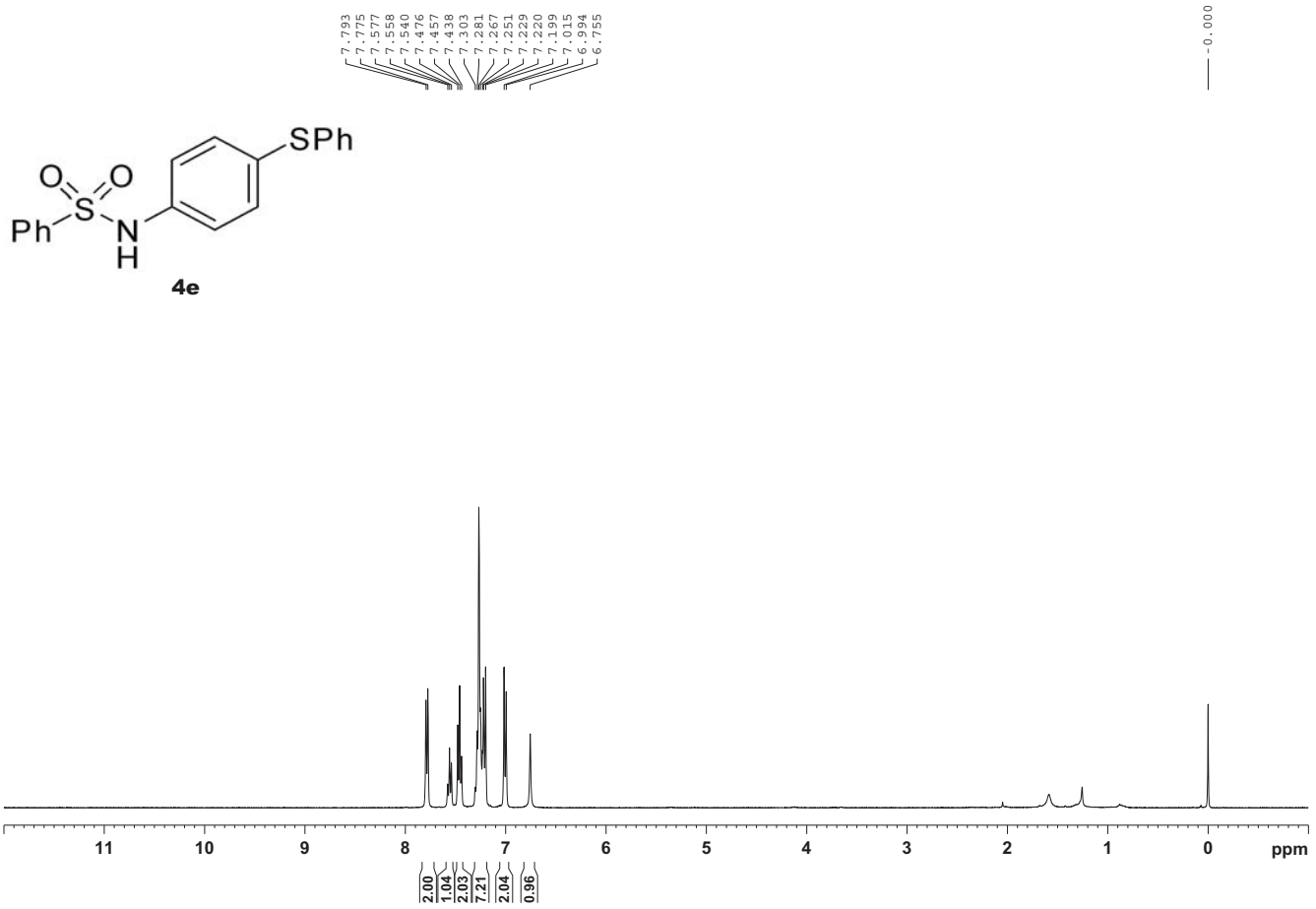


^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)



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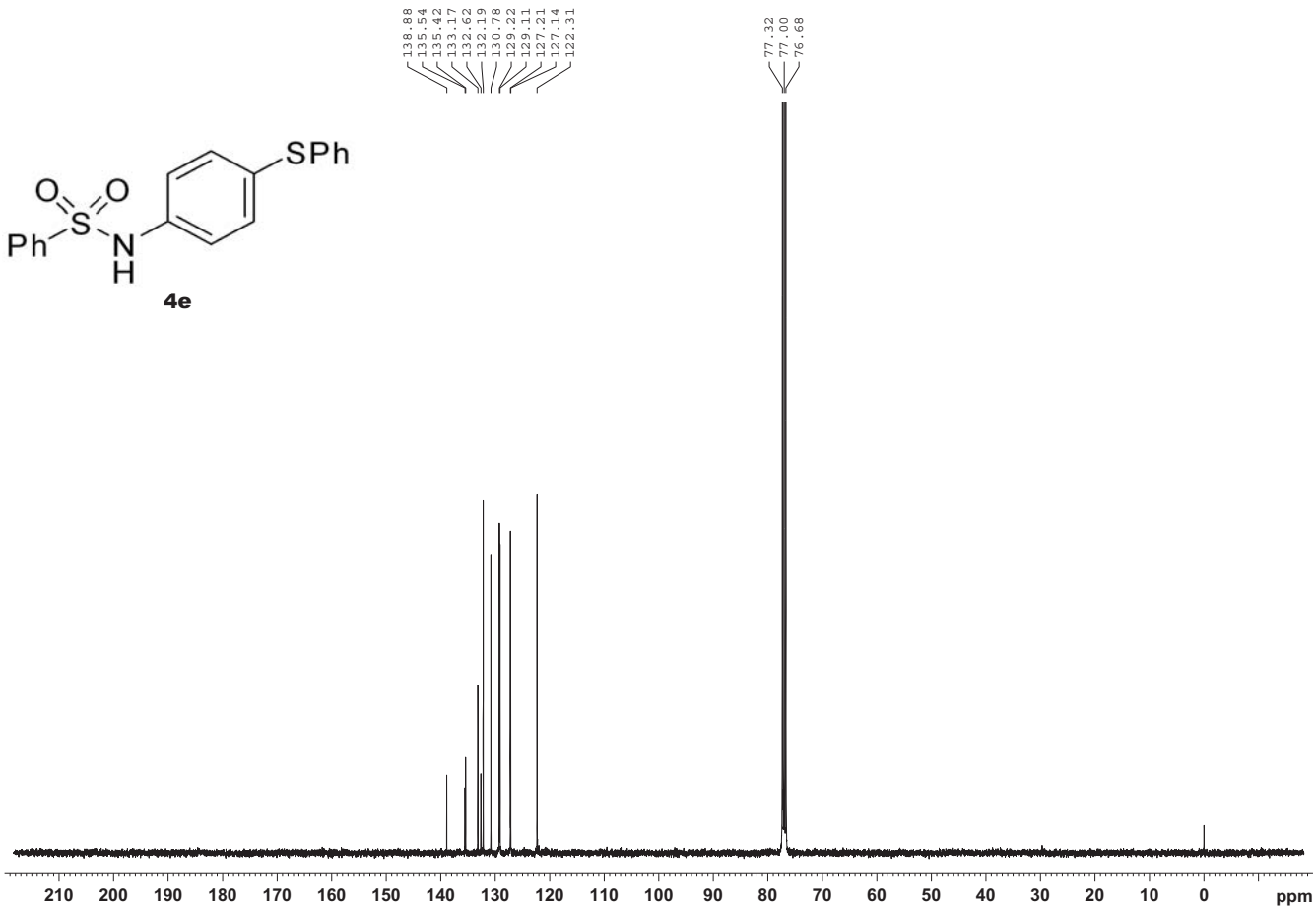
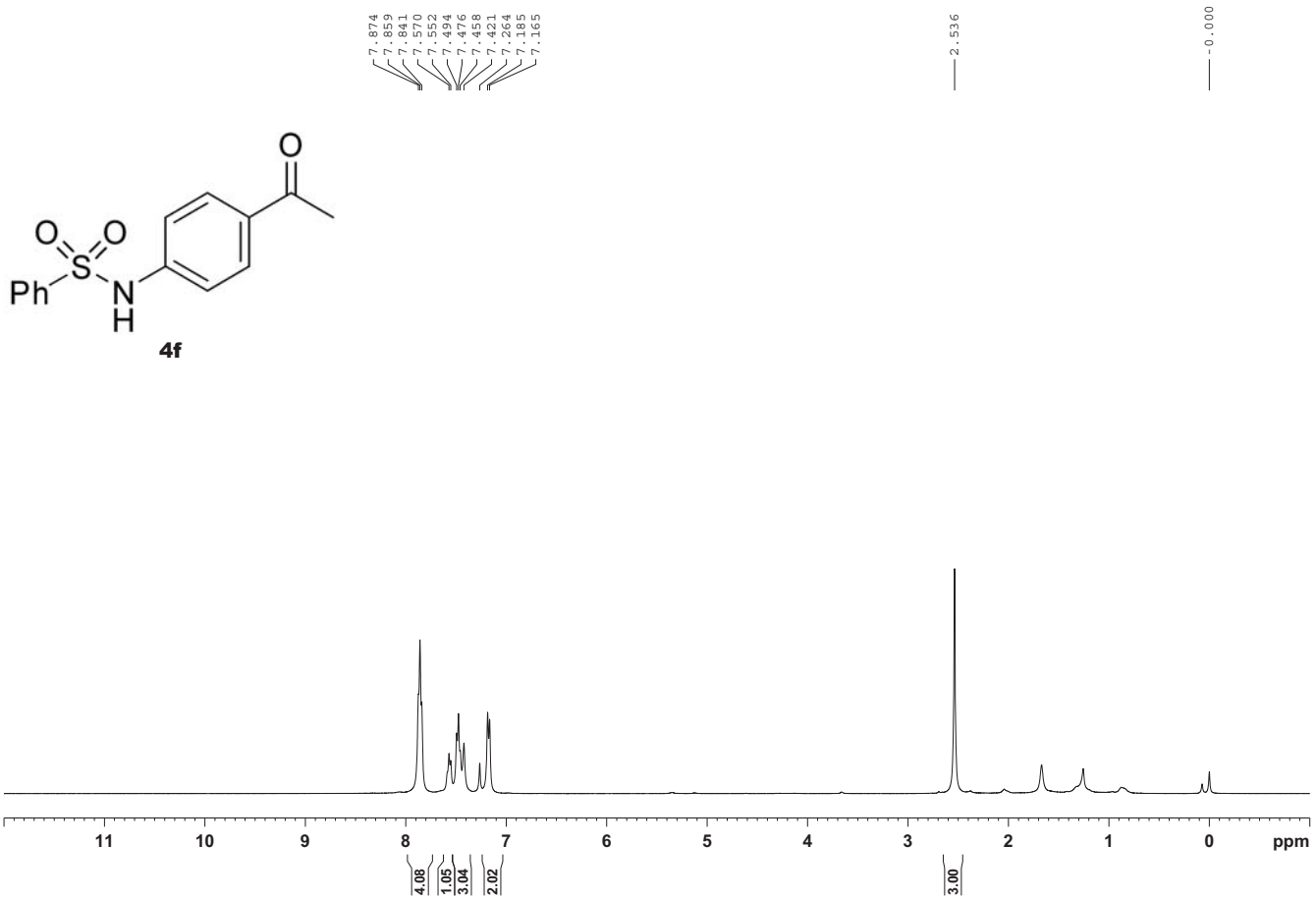
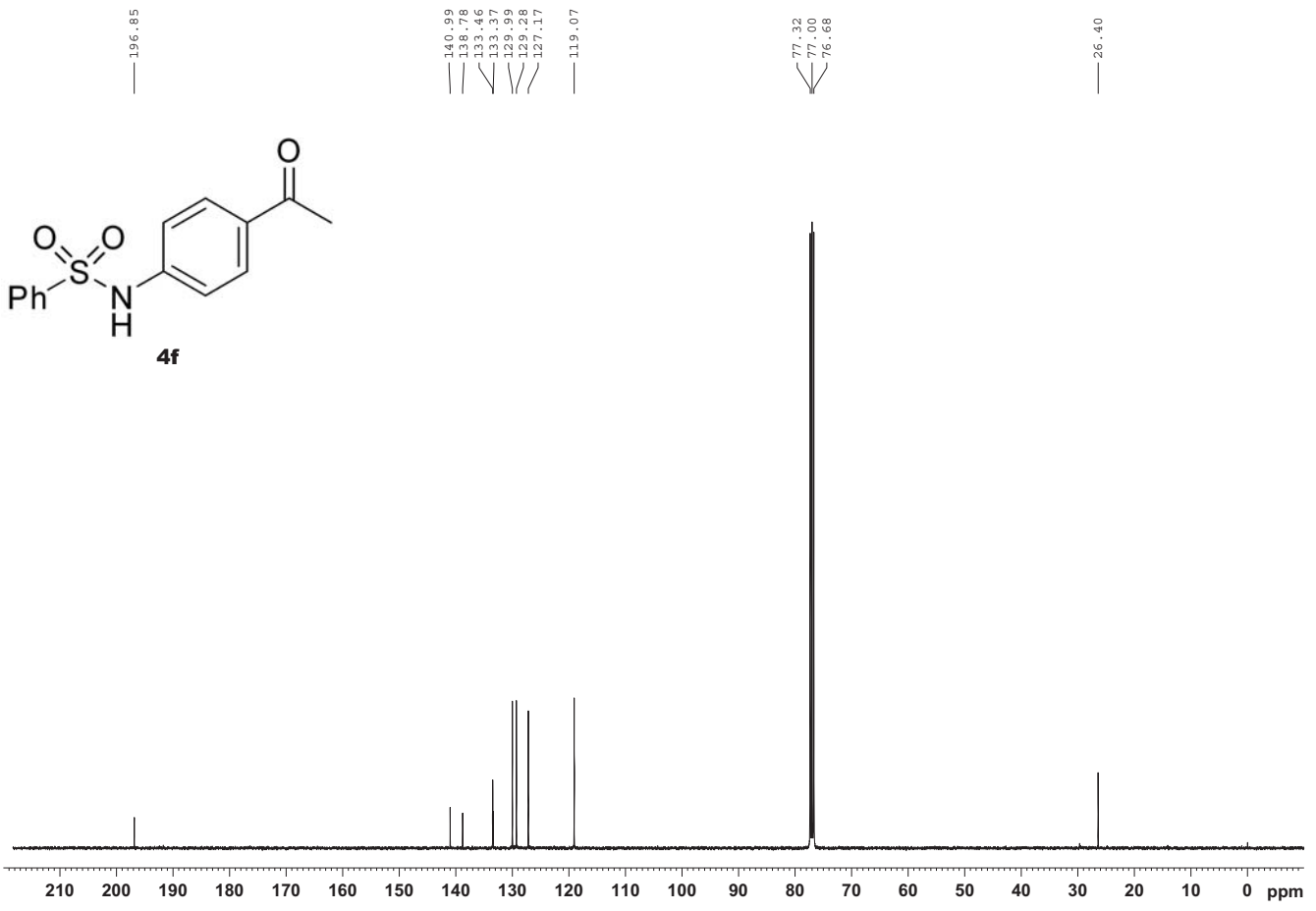


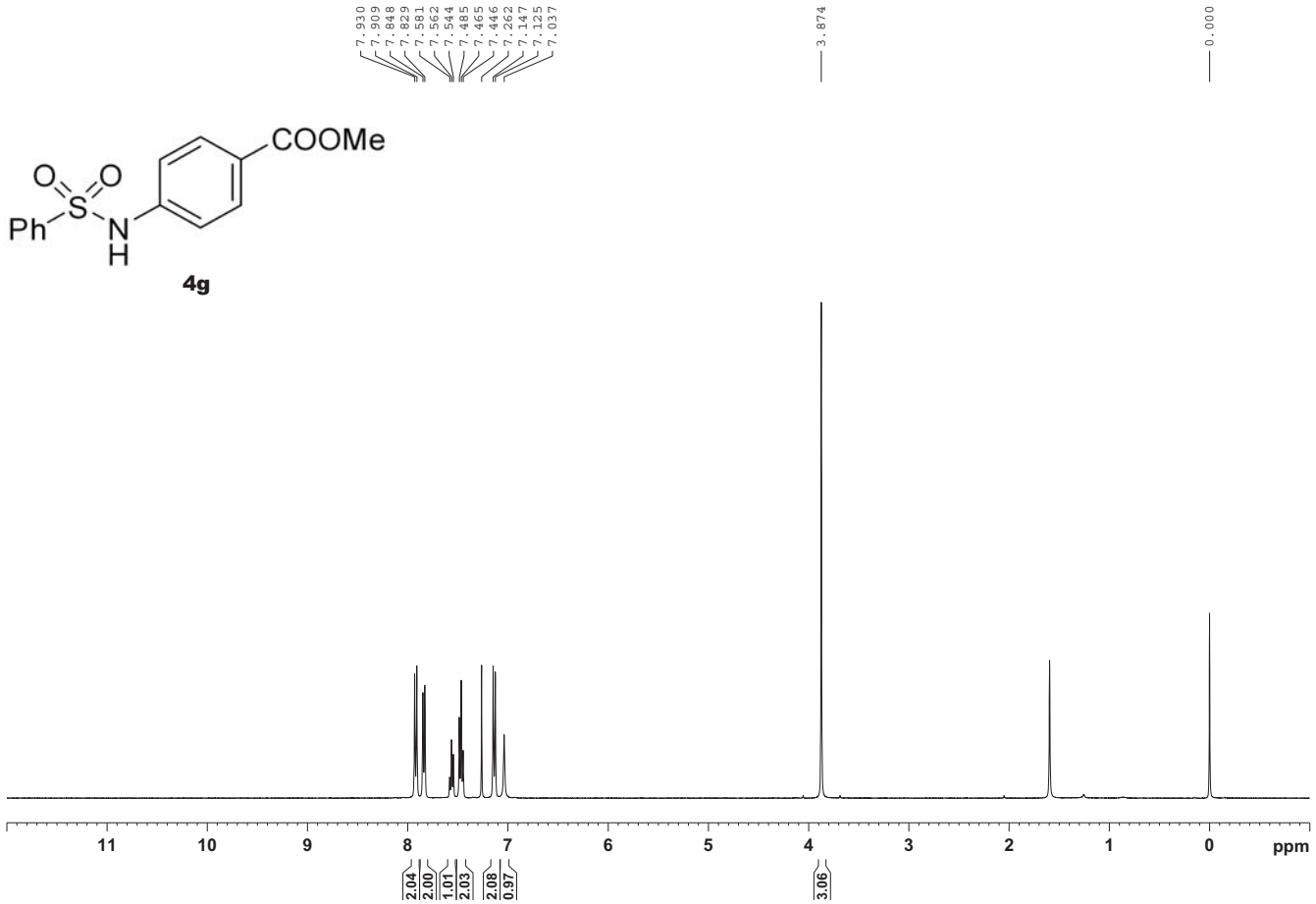
^{1}H NMR (400 MHz, $CDCl_3$)



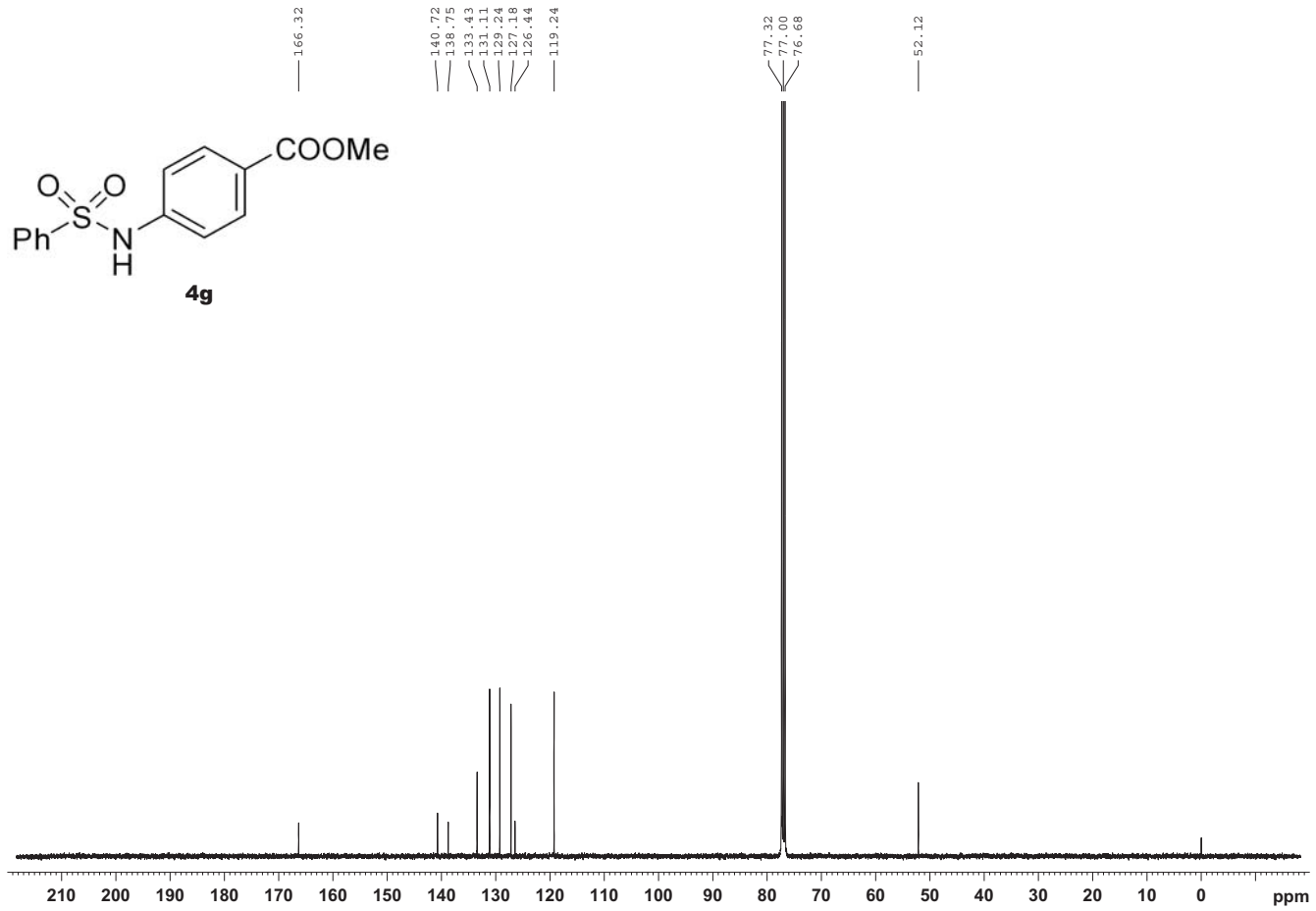
^{13}C NMR (101 MHz, $CDCl_3$)



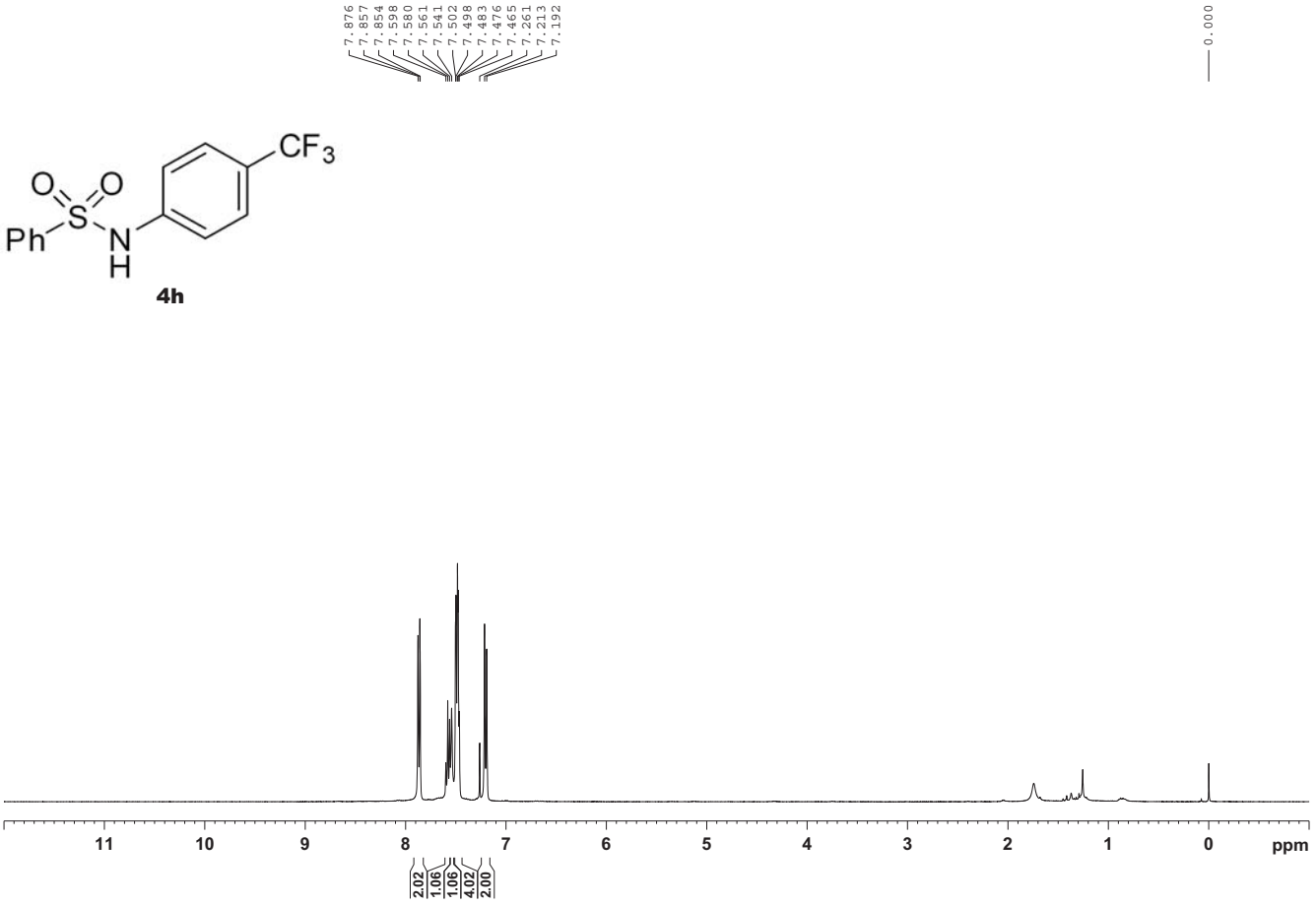
^{1}H NMR (400 MHz, $CDCl_3$)



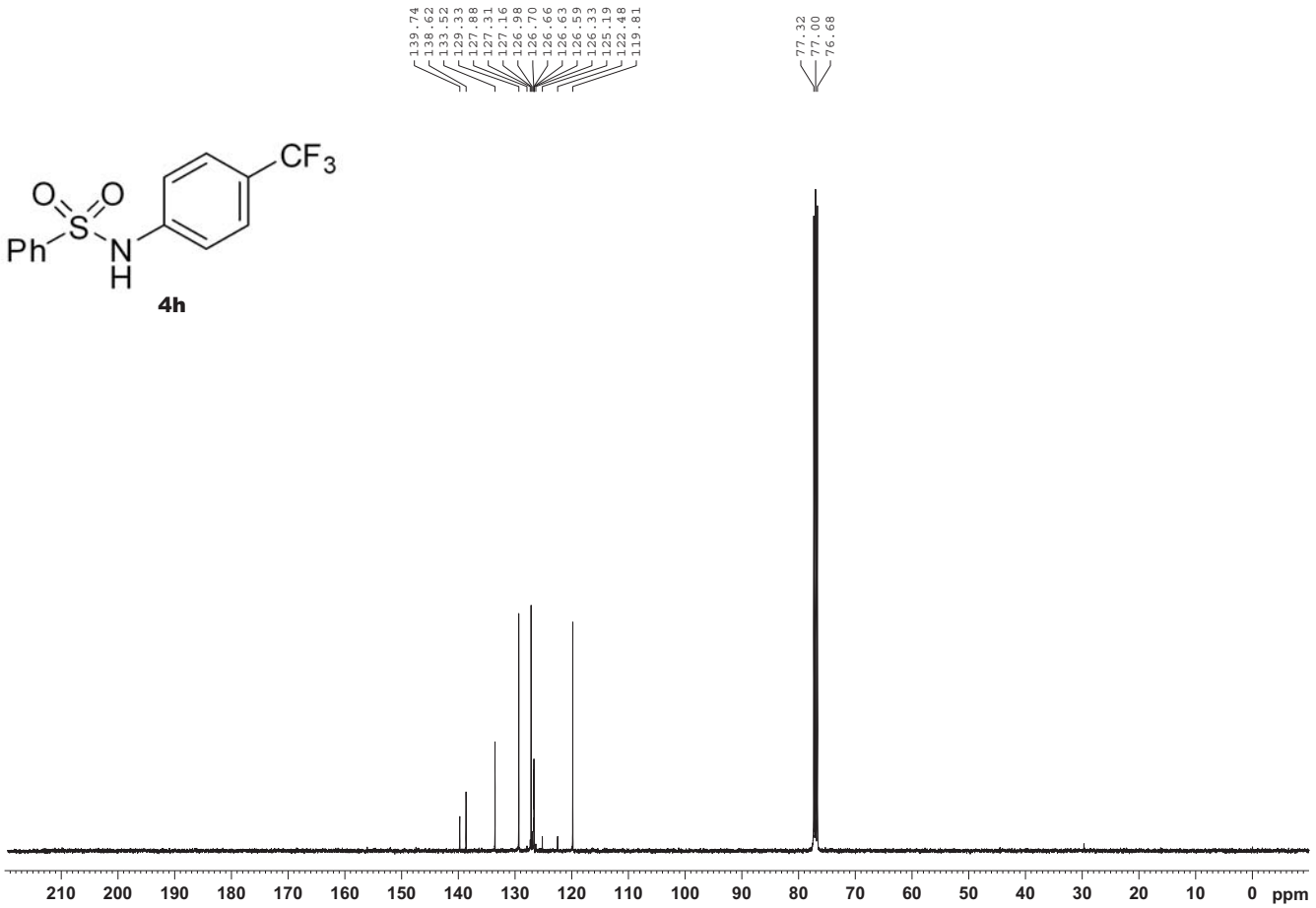
^{13}C NMR (101 MHz, $CDCl_3$)



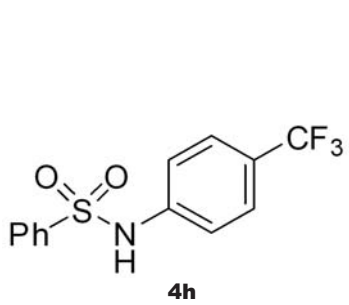
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)

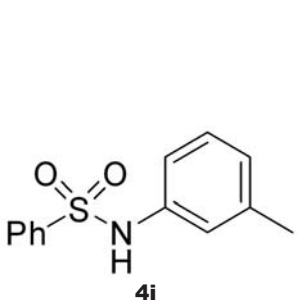


^{19}F NMR (376 MHz, $CDCl_3$)



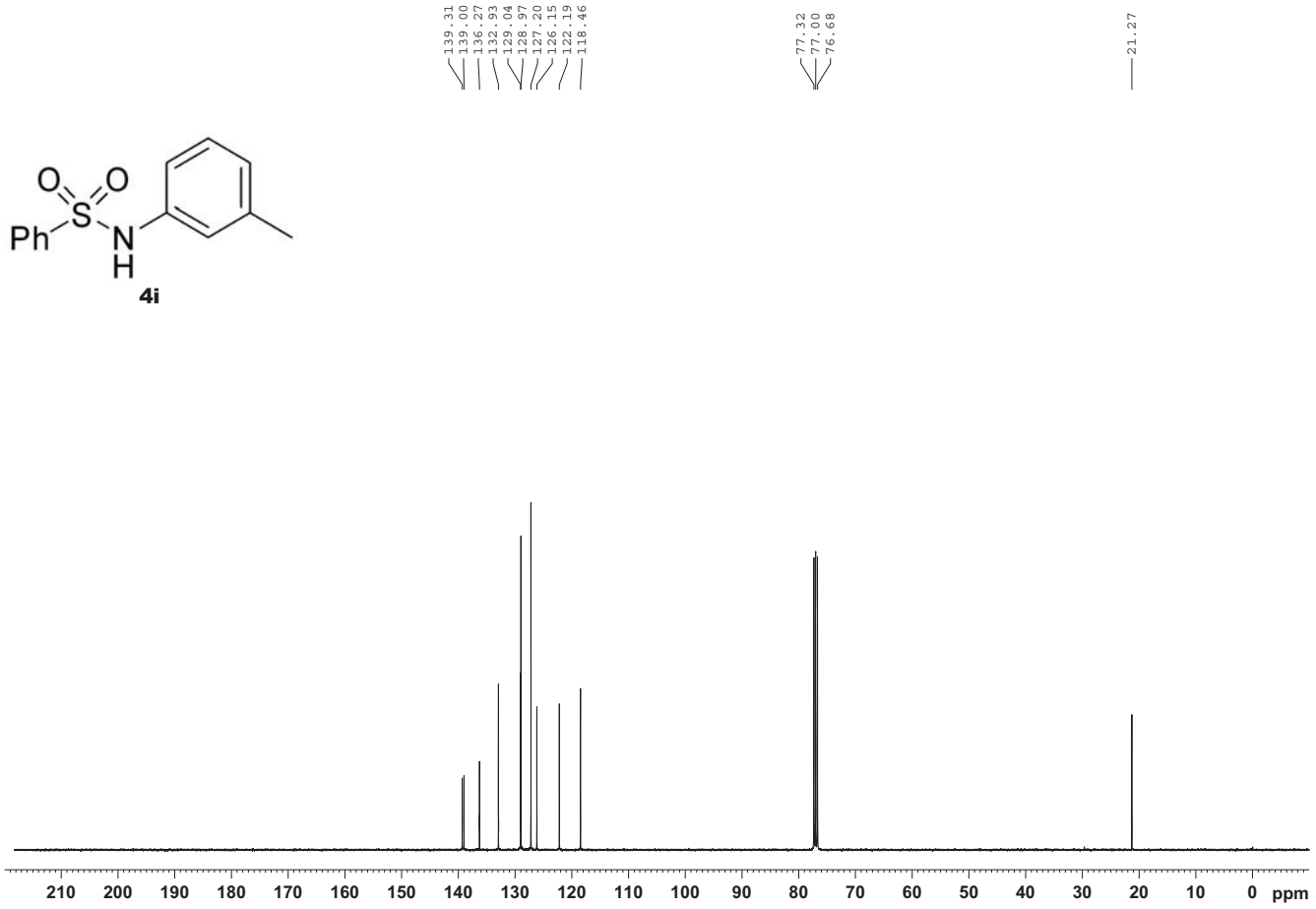
4h

^{1}H NMR (400 MHz, $CDCl_3$)

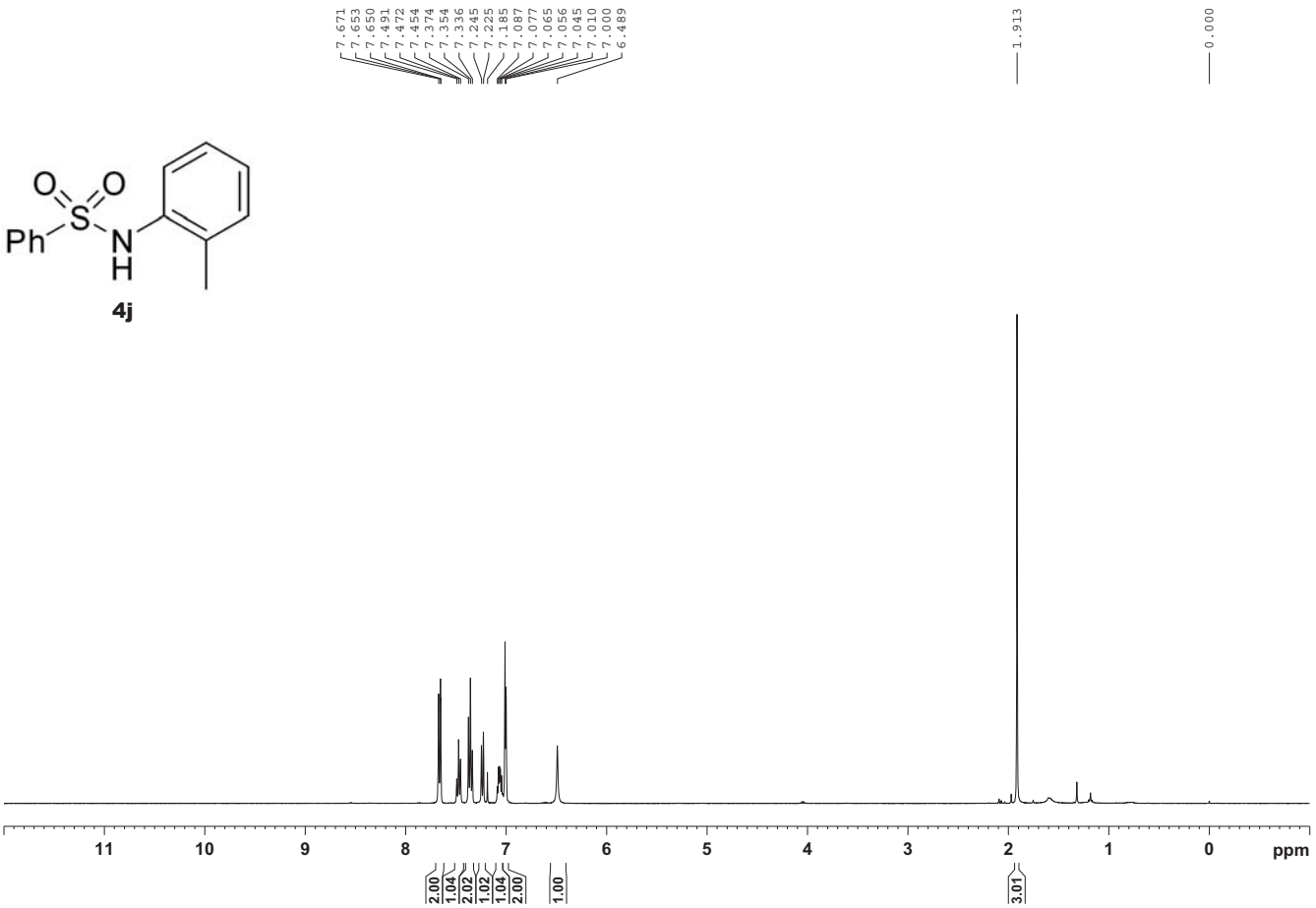


4i

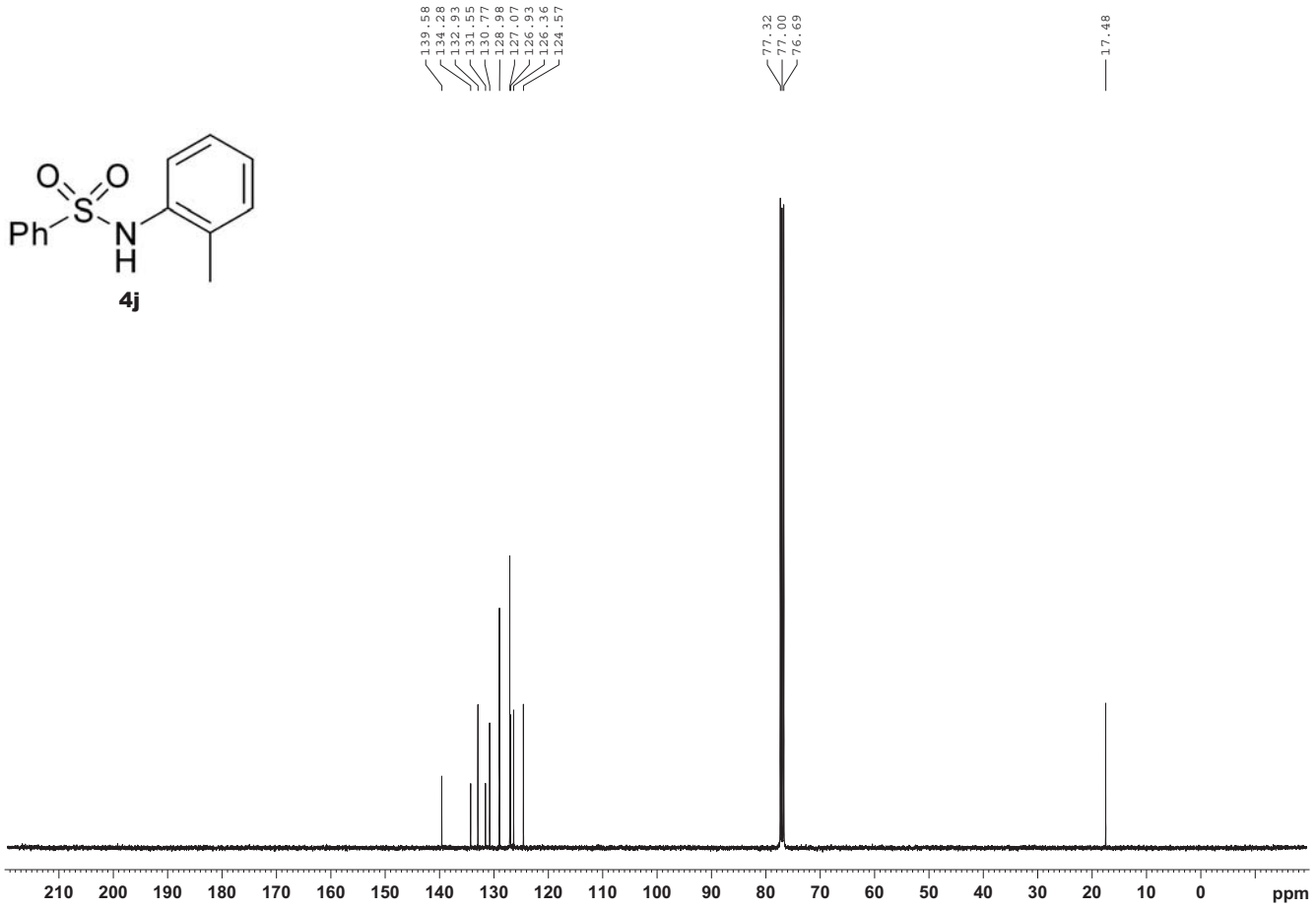
^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)

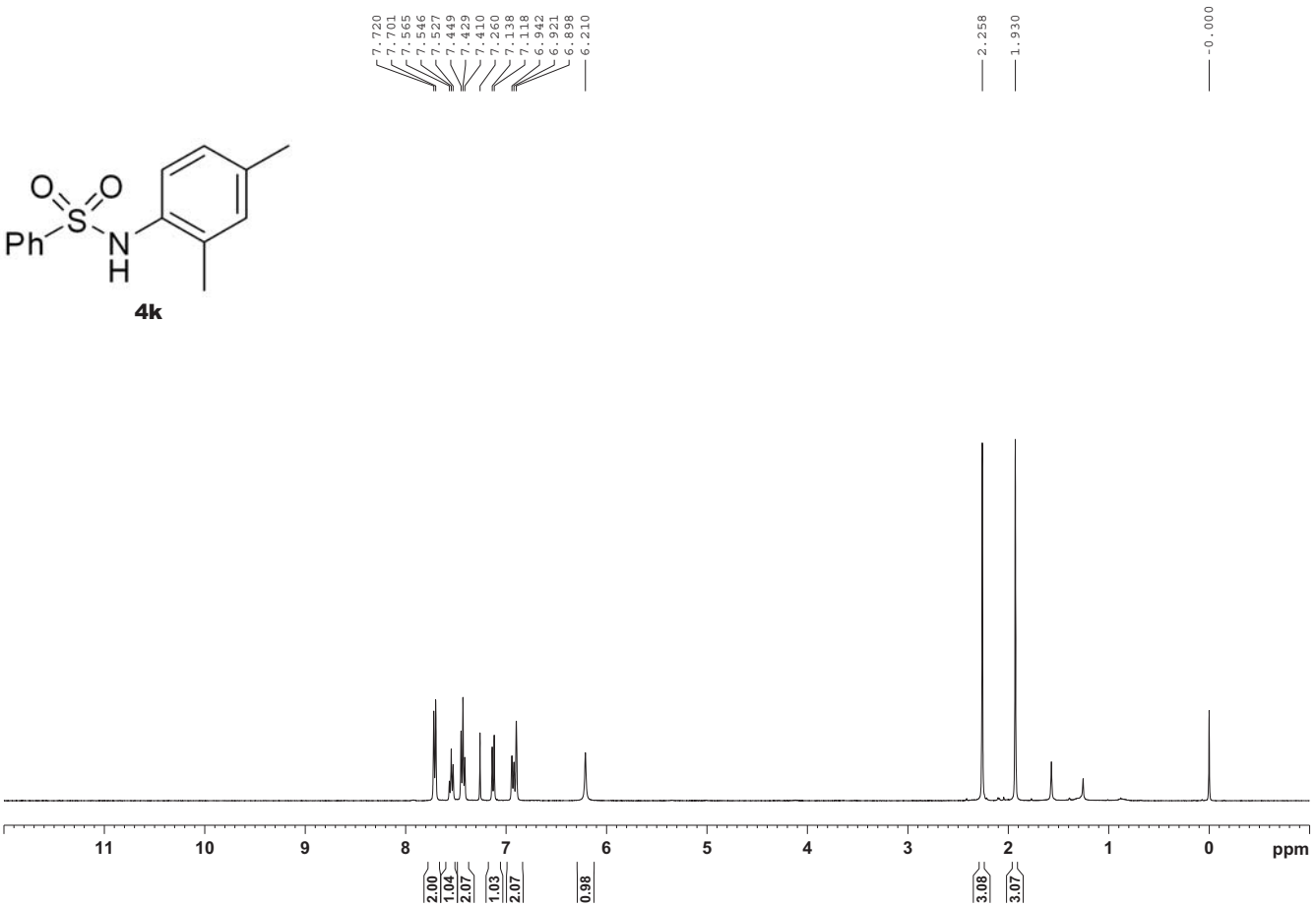


^{13}C NMR (101 MHz, $CDCl_3$)



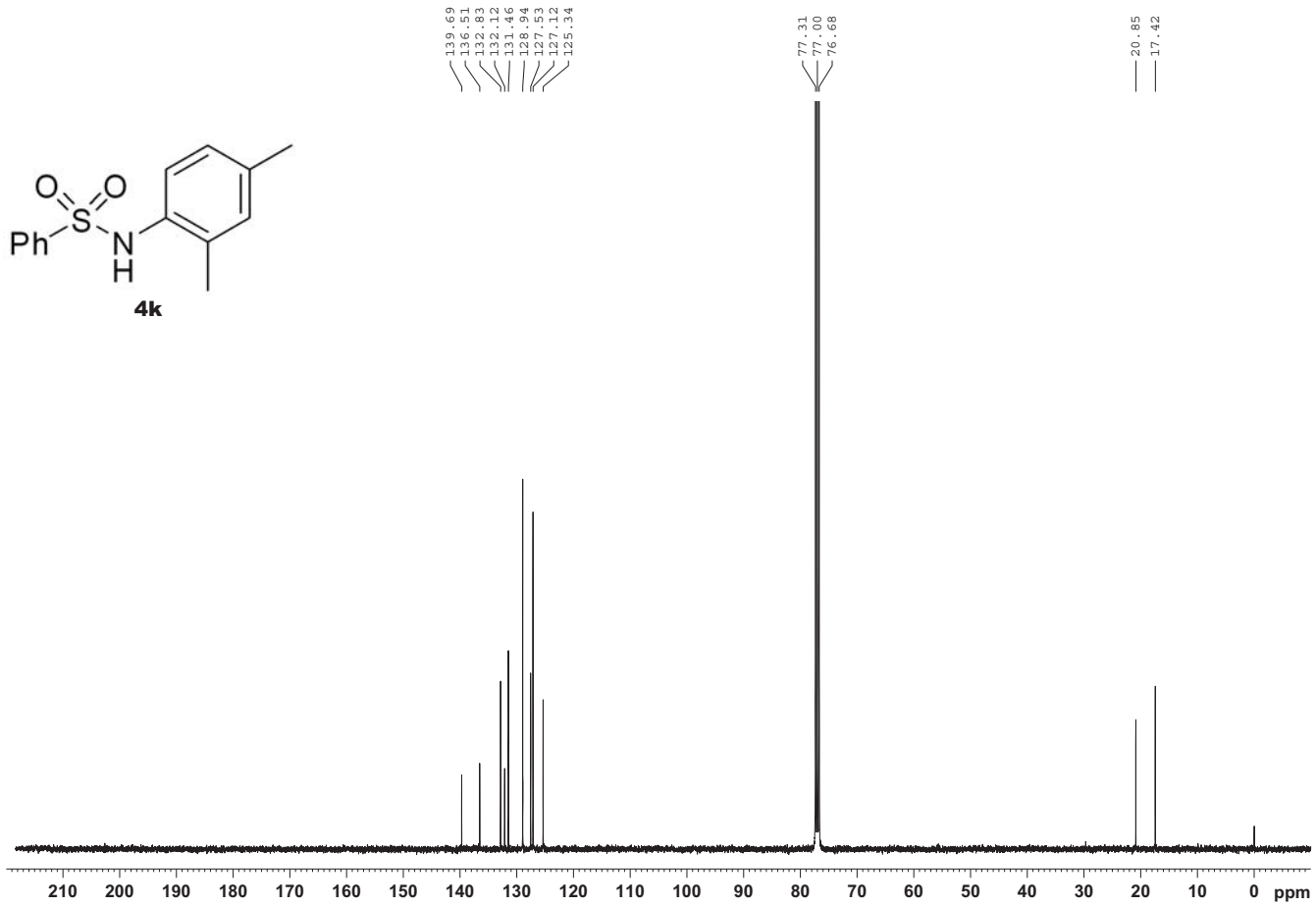
4j

^{1}H NMR (400 MHz, $CDCl_3$)

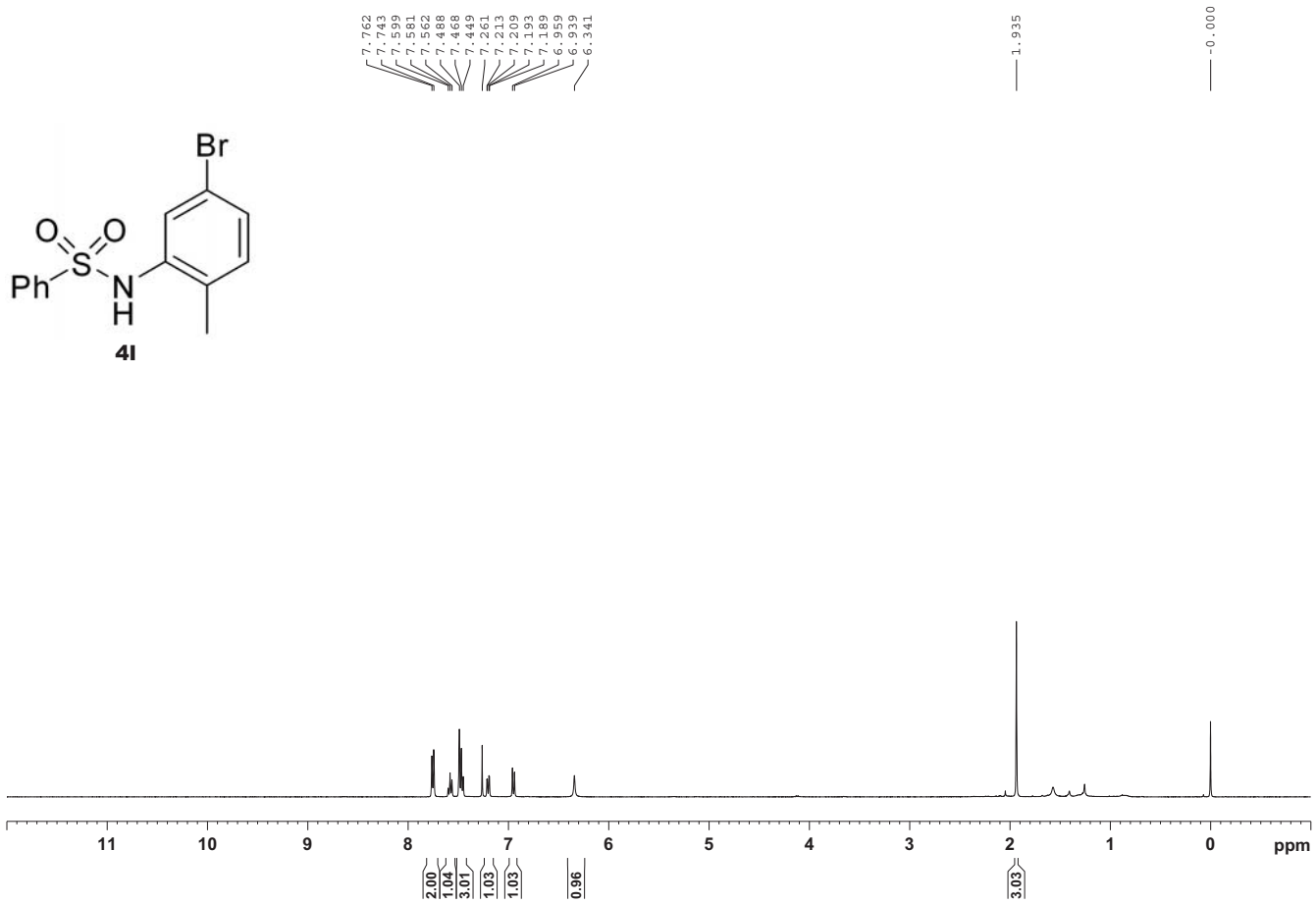


4k

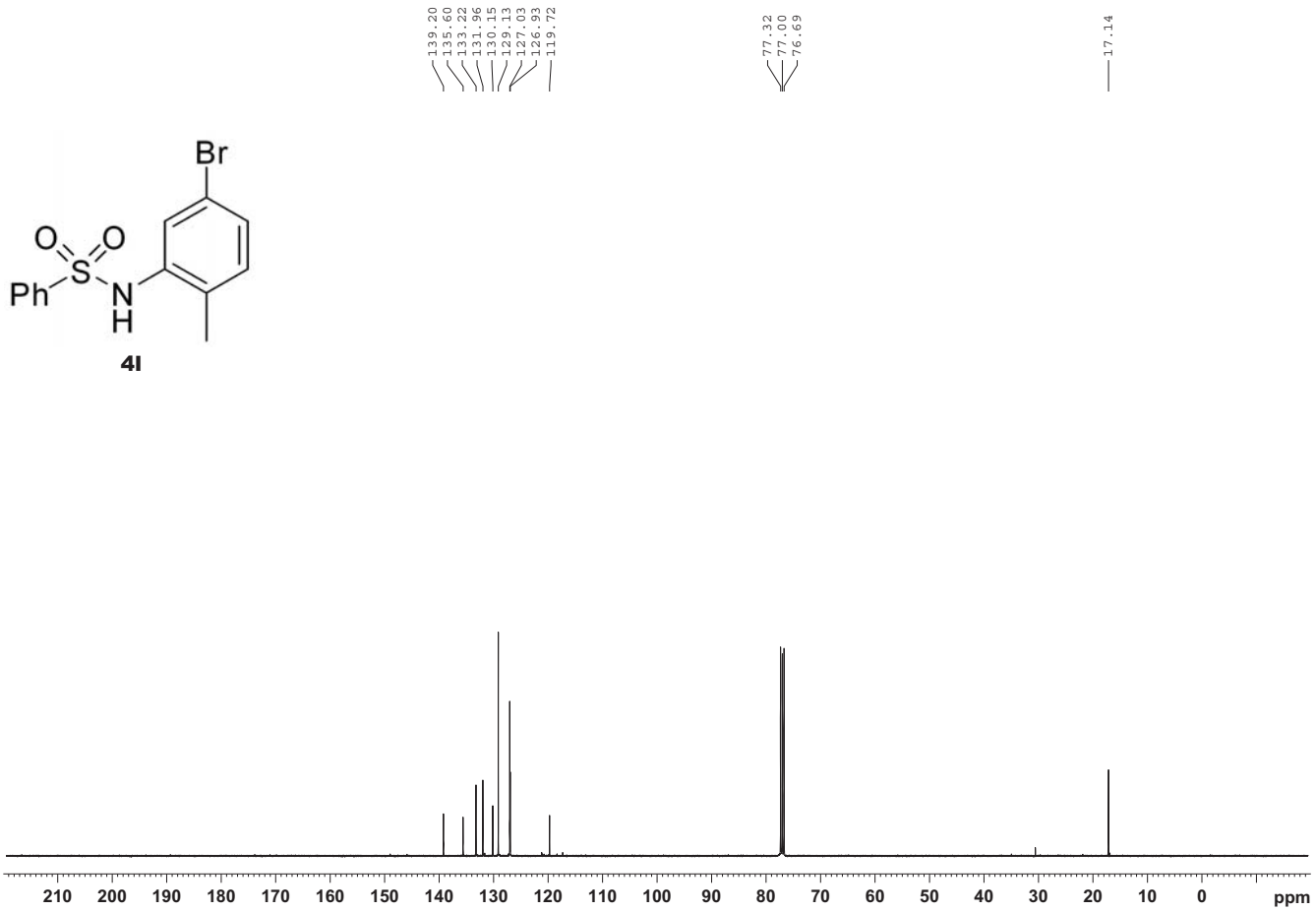
^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)

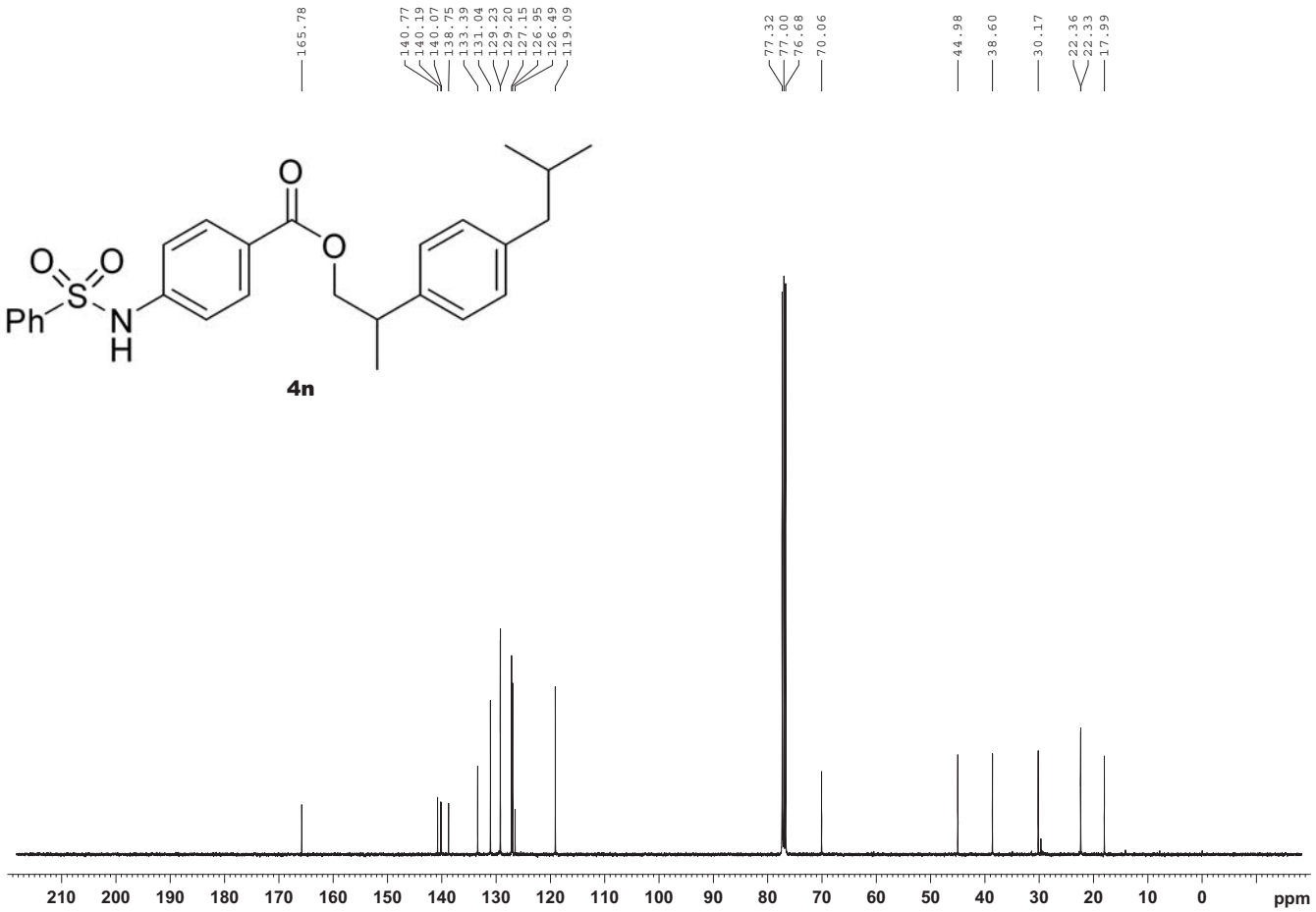


4l

^{1}H NMR (400 MHz, $CDCl_3$)

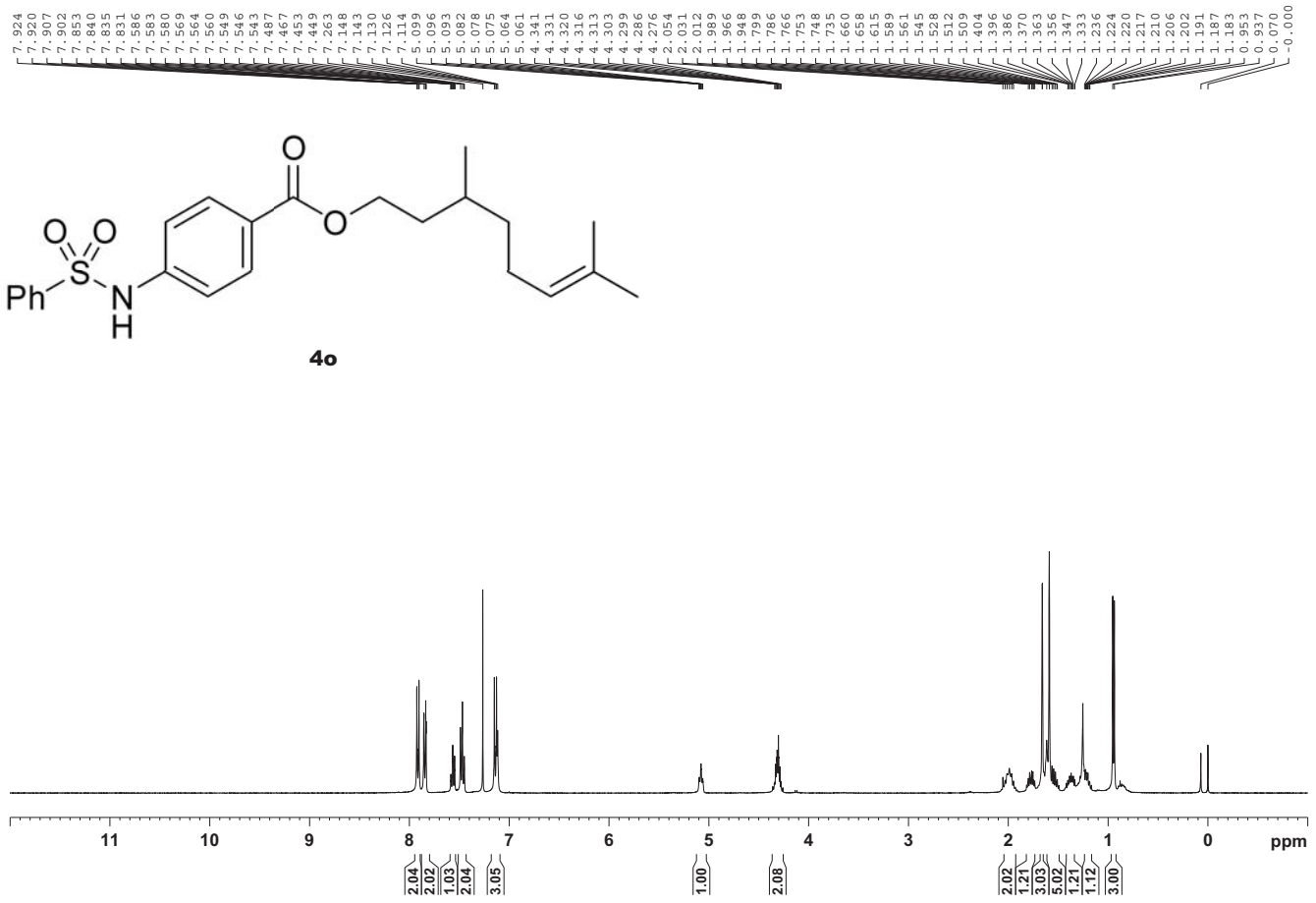
4n

^{13}C NMR (101 MHz, $CDCl_3$)



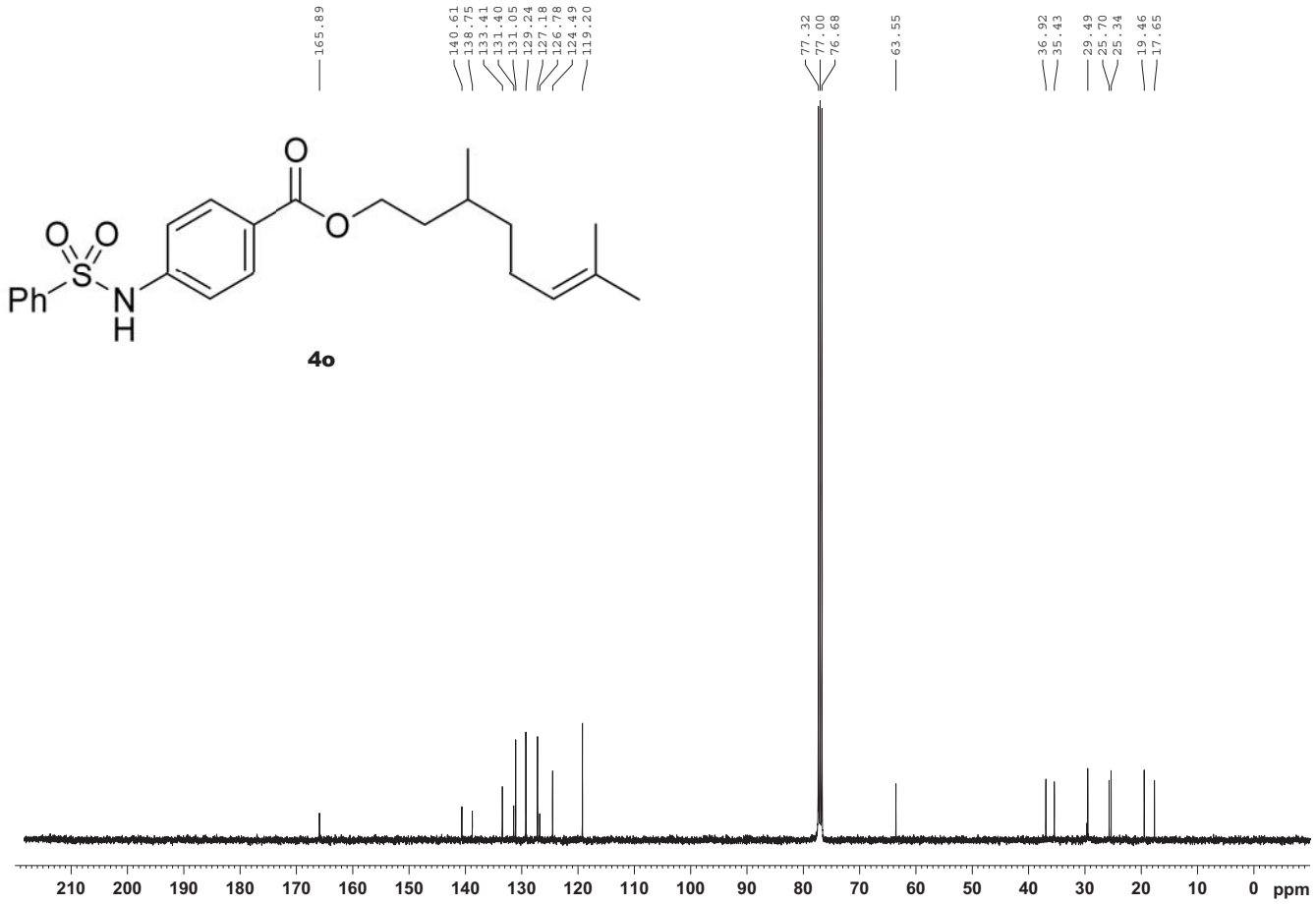
4n

^{1}H NMR (400 MHz, $CDCl_3$)



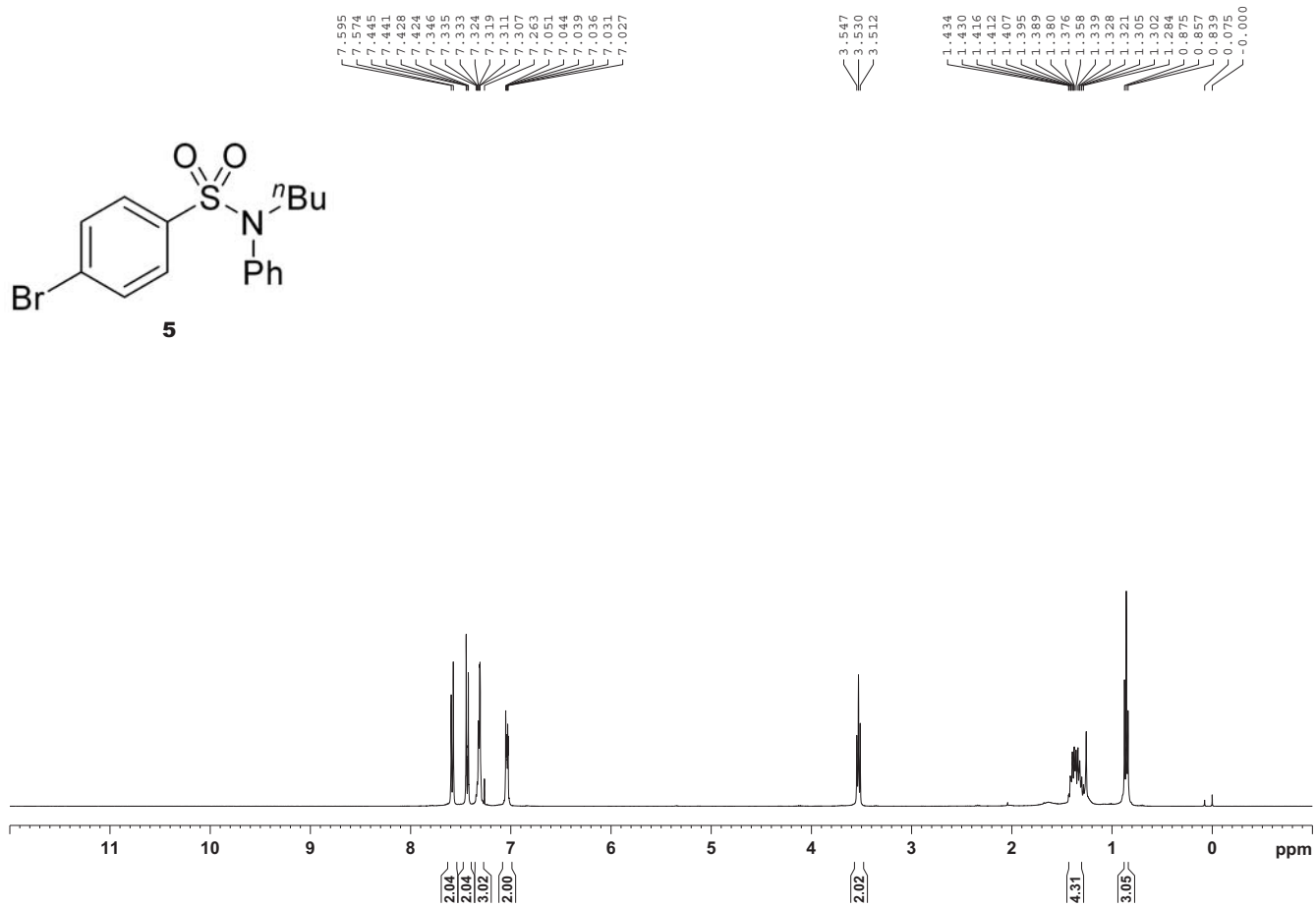
4o

^{13}C NMR (101 MHz, $CDCl_3$)



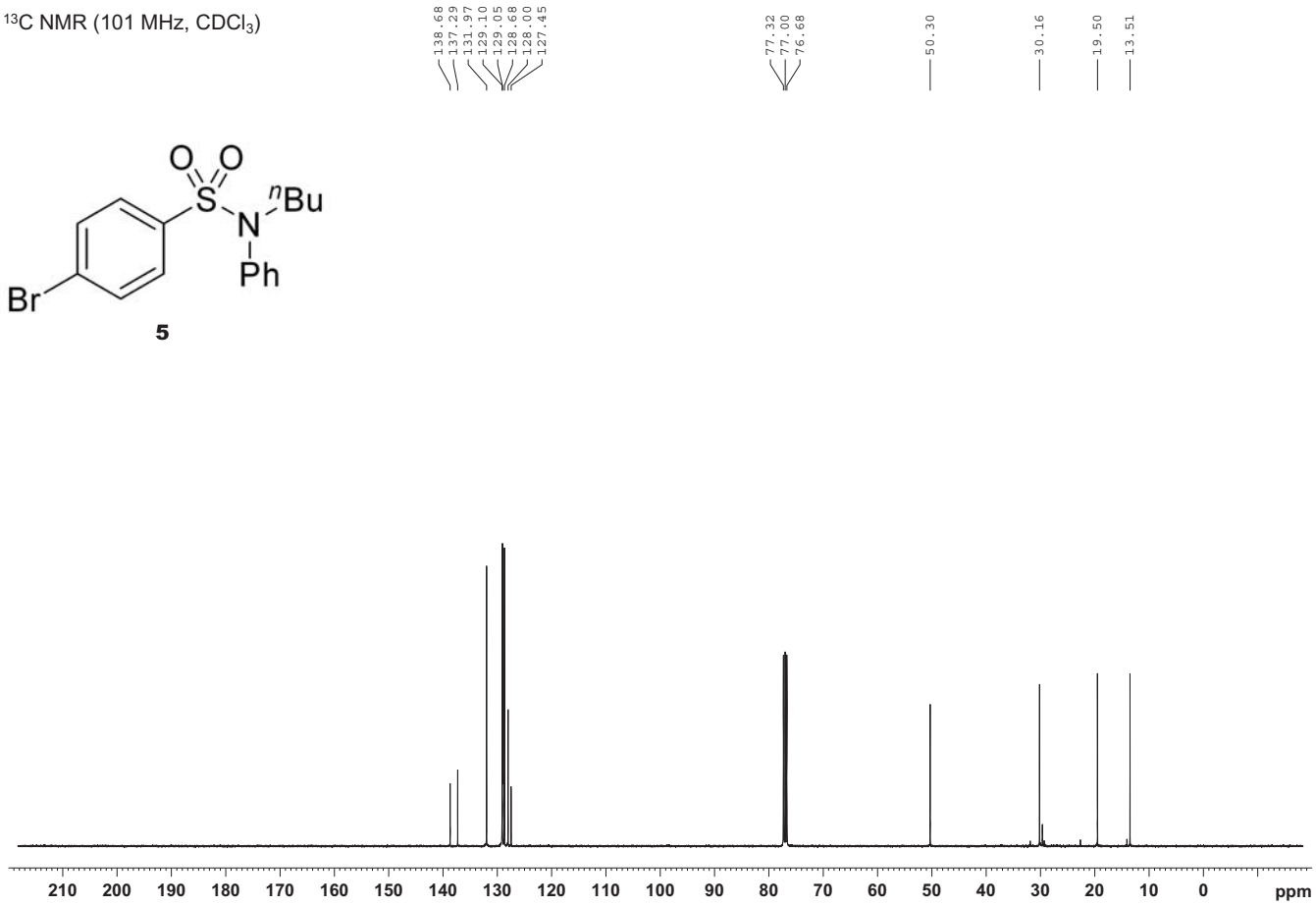
4o

^{1}H NMR (400 MHz, $CDCl_3$)



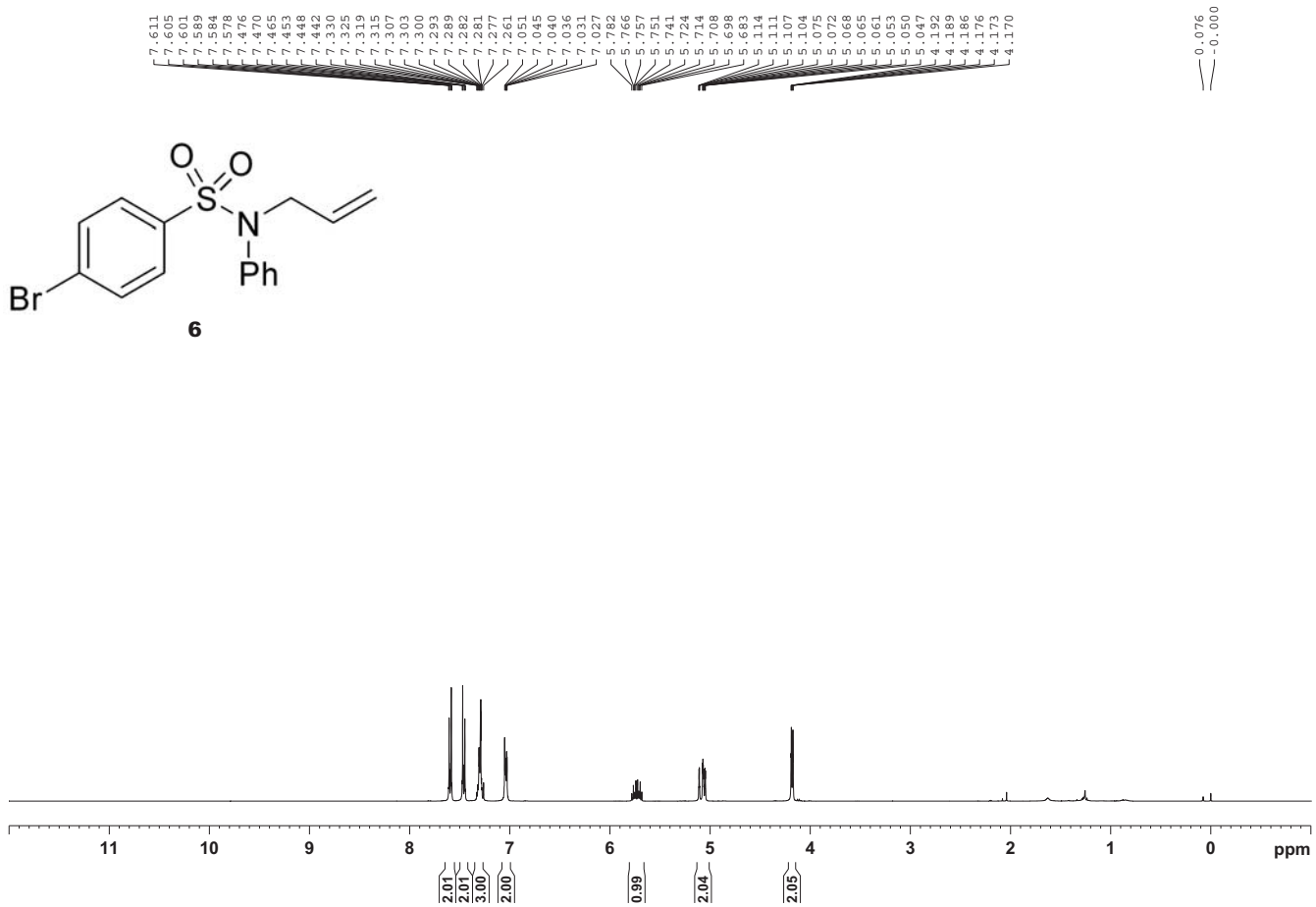
5

^{13}C NMR (101 MHz, $CDCl_3$)



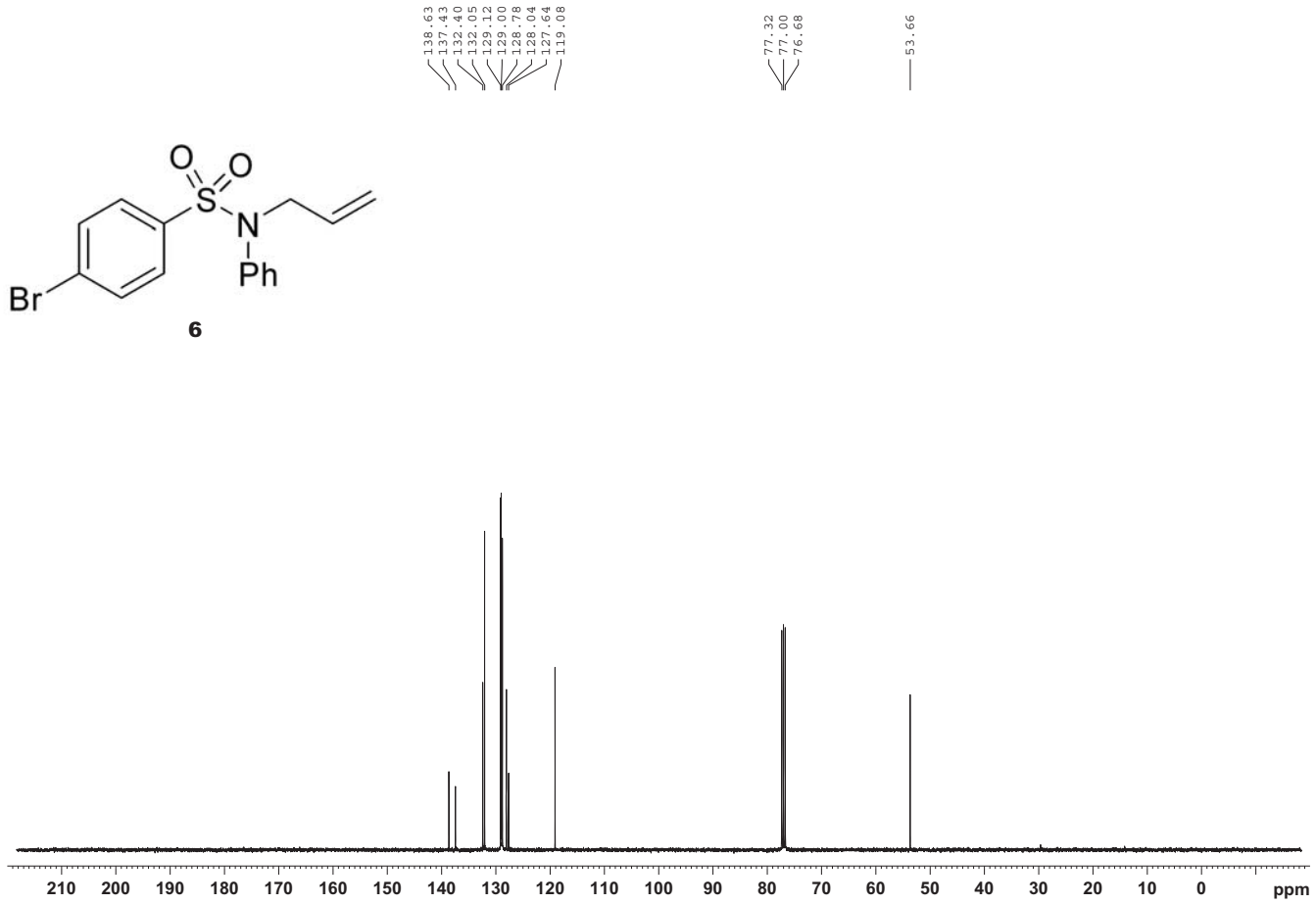
5

^{1}H NMR (400 MHz, $CDCl_3$)

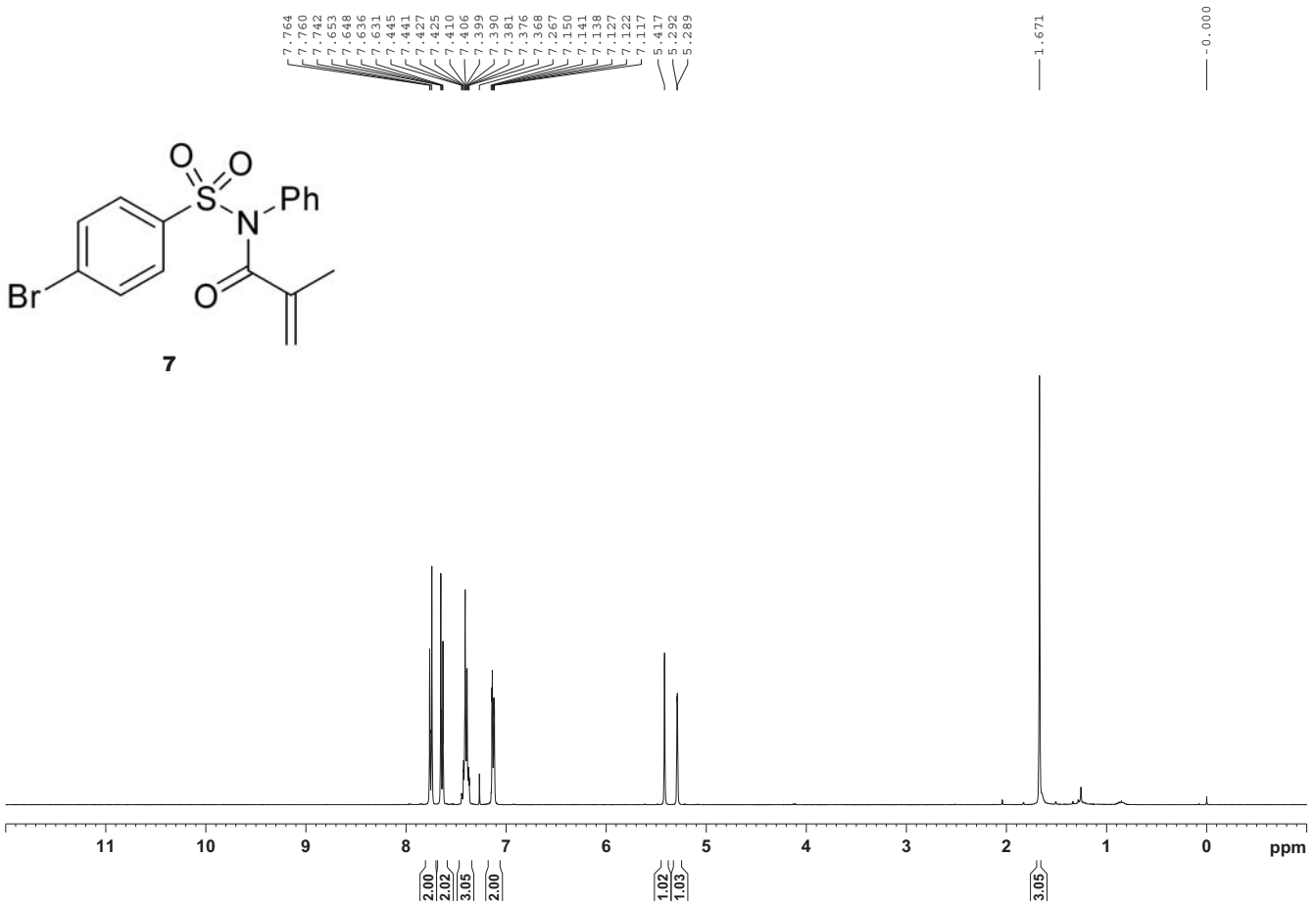


6

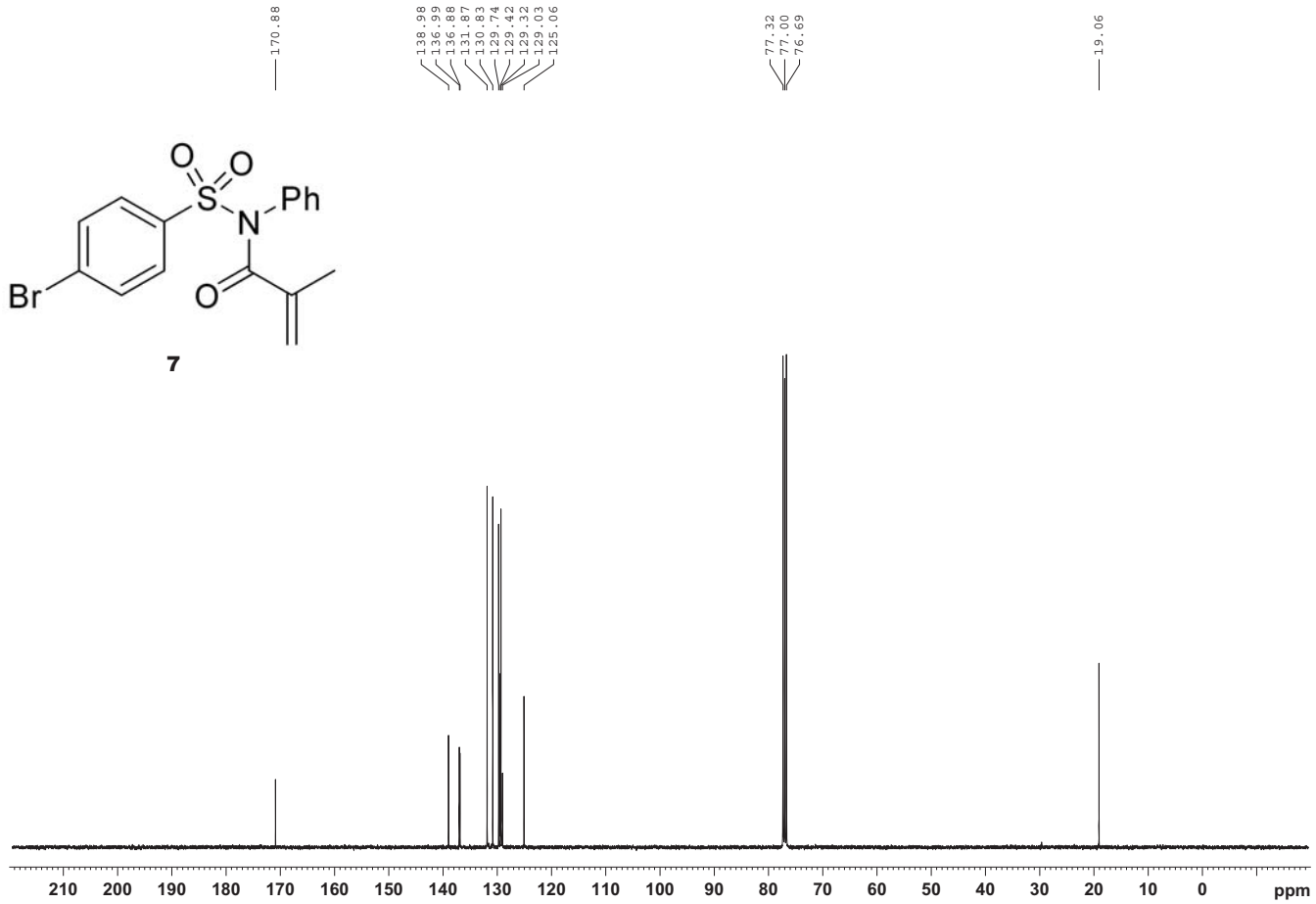
^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)

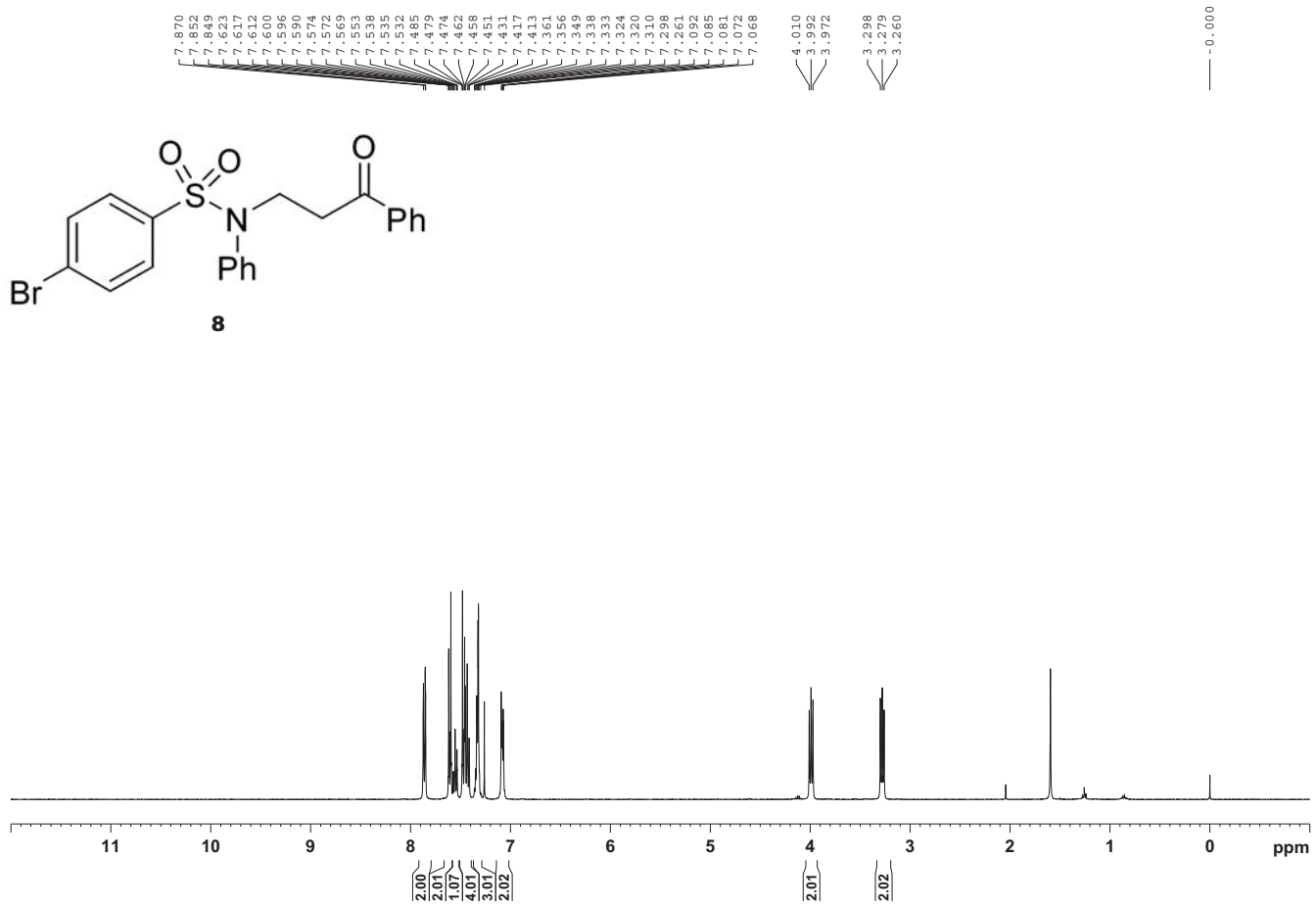


^{13}C NMR (101 MHz, $CDCl_3$)



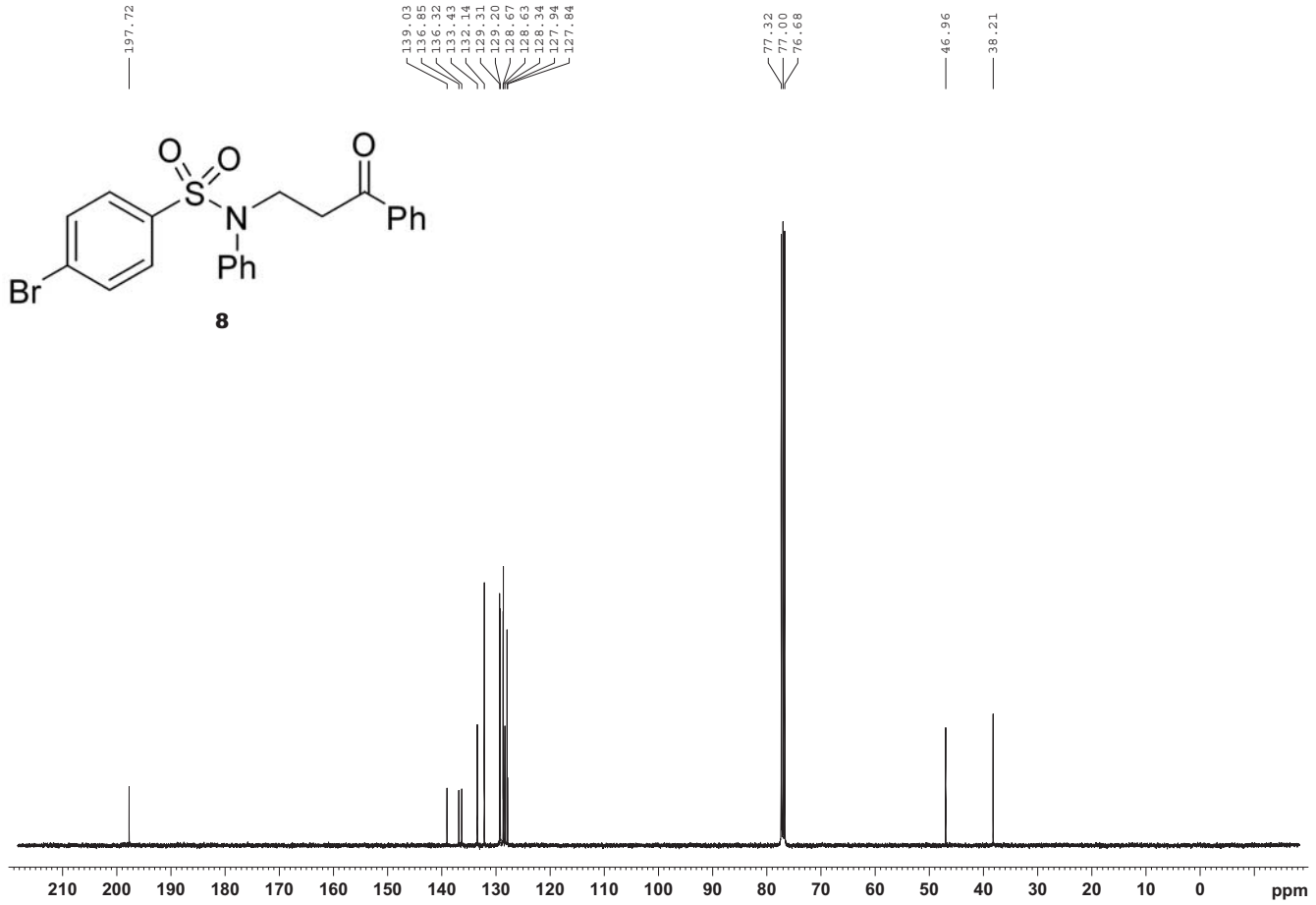
7

^{1}H NMR (400 MHz, $CDCl_3$)

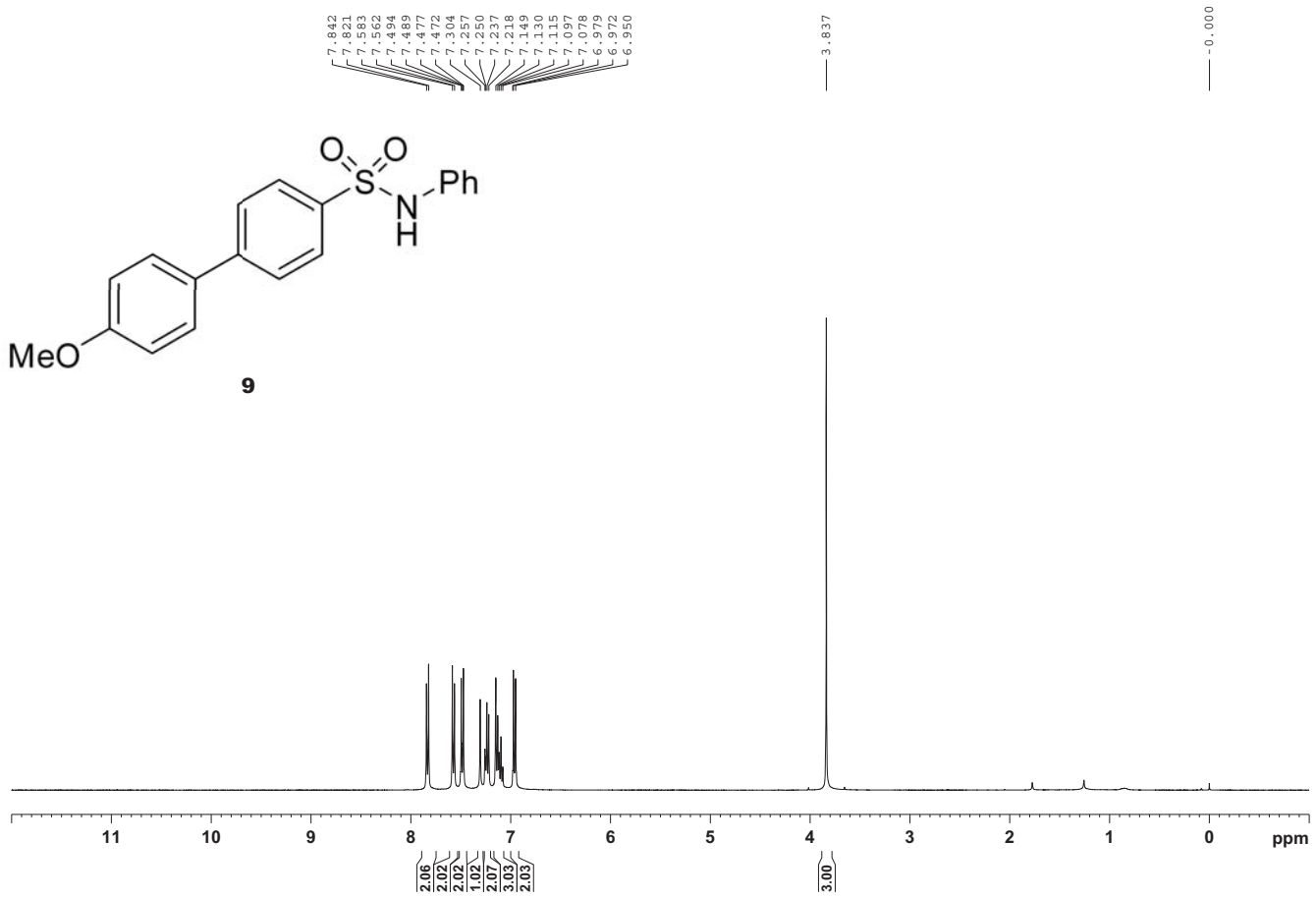


8

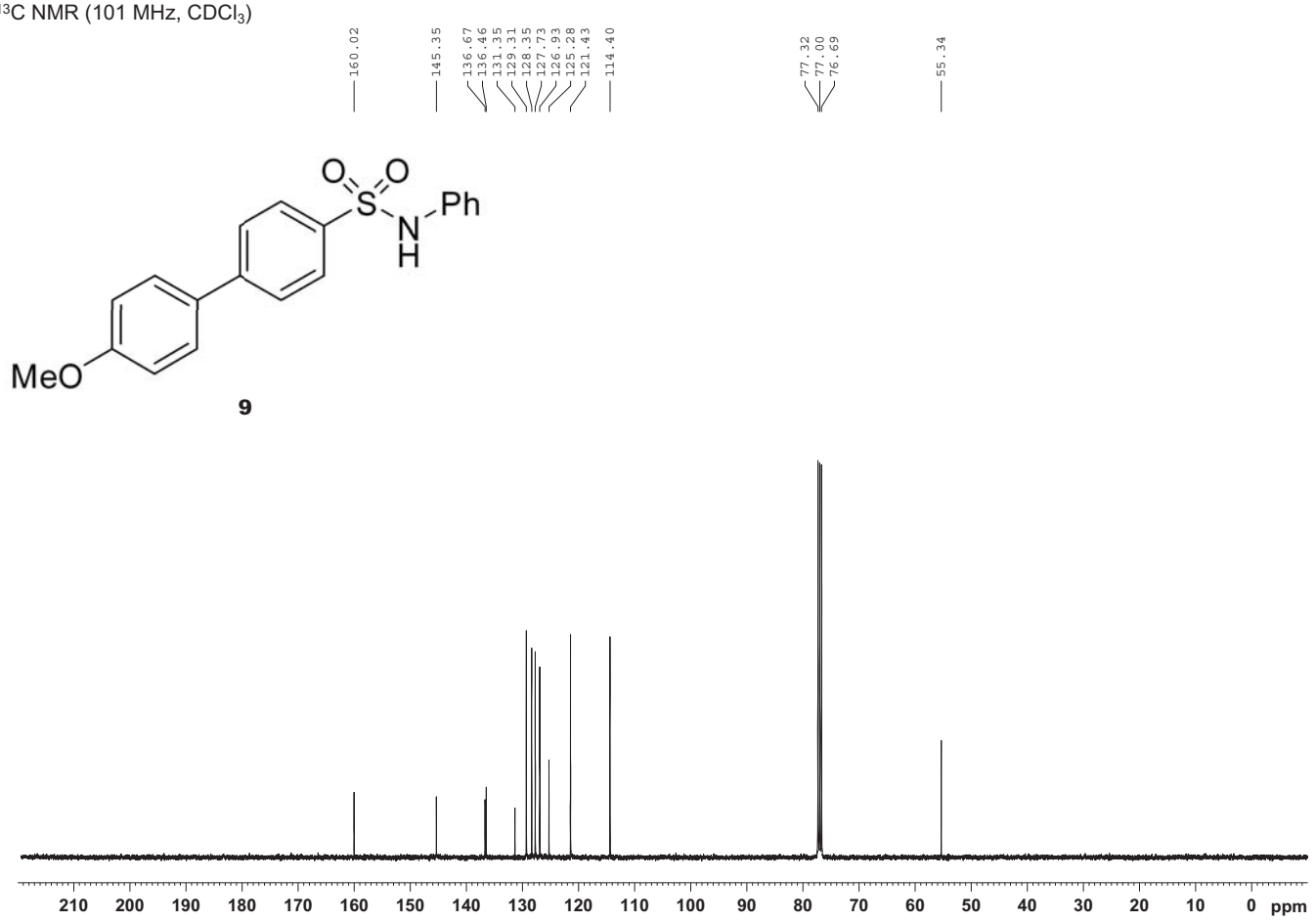
^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)

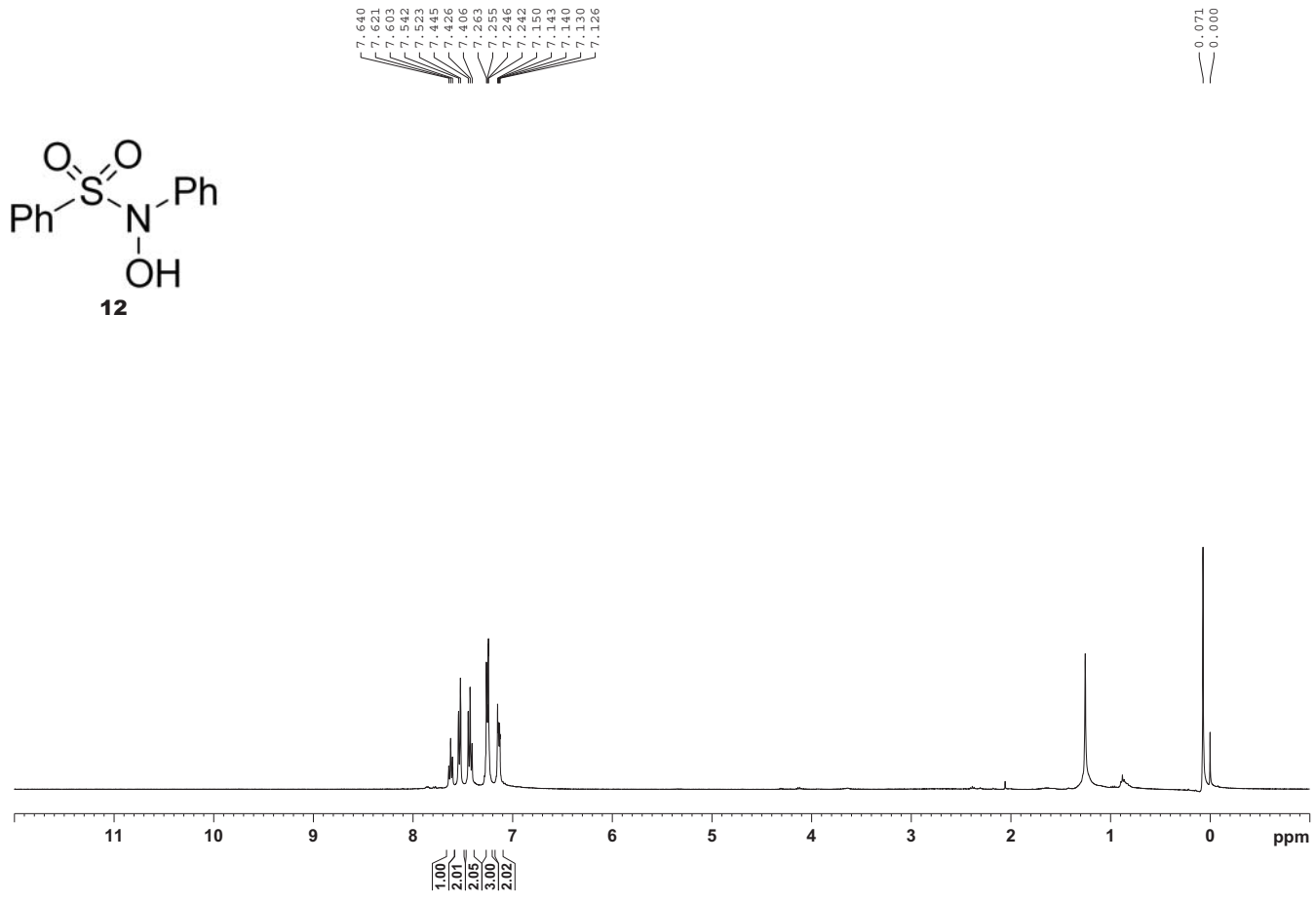


^{13}C NMR (101 MHz, $CDCl_3$)



9

^{1}H NMR (400 MHz, $CDCl_3$)



12

^{13}C NMR (101 MHz, $CDCl_3$)